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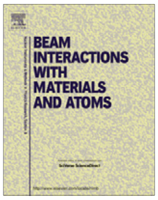

Carrier free ^{10}Be/^{9}Be measurements with low-energy AMS: Determination of sedimentation rates in the Arctic Ocean

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ABSTRACT

Using the TANDY AMS facility (600 kV) at ETH Zurich the seawater-derived (authigenic) ^{10}Be/^{9}Be ratio of marine sediment samples is measured without the addition of ^{9}Be carrier. This novel method reduces systematic uncertainties because the ^{10}Be/^{9}Be ratio of a sample is determined in only one (AMS) measurement. A challenge of carrier-free AMS is to avoid any contamination of the sample with ^{9}Be during the chemical preparation. Further, the leaching procedure has to be reproducible and ideally should attack the authigenic Be of the sediments only, leaving the detrital Be untouched. The low amount of stable ^{9}Be in the unspiked samples causes low currents during the AMS measurement. This requires a good stability and sensitivity of the AMS setup. Our first results show that the new preparation method is reliable and that background from stable ^{9}Be is avoided.

For a comparison study, sediment samples from two cores located in the Arctic Ocean (HLY0503-09JPC, HLY0503-14JPC) were used. The authigenic ^{10}Be/^{9}Be ratio of these samples had been determined previously applying the conventional method where ^{10}Be and ^{9}Be concentrations are measured separately by AMS and ICP-MS, respectively. The resulting sedimentation rates are in discrepancy with values derived from biomarkers. To cross check the ^{10}Be/^{9}Be based age model two samples from each core were measured again with the new carrier-free method. The carrier-free results show systematically higher authigenic ^{10}Be/^{9}Be ratios. The calculated sedimentation rates of about 0.2 cm/kyr, however, are consistent for the carrier free and the conventional method.

1. Introduction

The authigenic (bottom-seawater-derived) ^{10}Be/^{9}Be ratio was early suggested for applications like dating marine archives [1] and studying geomagnetic field variations [2]. The conventional method to determine this ratio is measuring the ^{10}Be concentration with Accelerator Mass Spectrometry (AMS) and the ^{9}Be concentration with an appropriate method for abundant stable isotopes. However, the authigenic ^{10}Be/^{9}Be ratio can also be determined directly with AMS. Previously, carrier-free ^{10}Be/^{9}Be measurements with AMS were undertaken at the ETH Zurich using a SIMS source [3] or low-energy AMS [4].

In the first part of this paper, the sample processing and the instrumental settings of the carrier-free ^{10}Be AMS measurement at the 600 kV TANDY are described. In addition, results from multiple processings of three marine sediments document the reproducibility of the chemical procedure. The remeasurement of four sediment samples from the Arctic Ocean tests if the results of the new carrier-free method are consistent with values determined in a previous conventional ^{10}Be/^{9}Be measurement [5]. Further, the sedimentation rates in the Arctic Ocean calculated from the ^{10}Be/^{9}Be ratio are compared with values derived from biomarkers and from ^{231}Pa/^{230}Th.

2. Methods

2.1. Sample description

Three marine sediment samples named (1021-8, 1021-12 and 1021-15) from the Ocean Drilling Program Site 1021 at the California margin [6] were used to check the reproducibility of the chemistry and the AMS measurement by multiple preparations and measurements of the same sample material.

During the 2005 HOTRAX Expedition to the Arctic Ocean a number of sediment cores were taken [7]. Two of them, HLY0503-09JPC (09JPC) at the Mendeleev Ridge (2783 m water depth, 15.4 m core length) and HLY0503-14JPC (14JPC) at the Alpha Ridge (1856 water depth, 11.2 m core length) had been analyzed for their authigenic Be content and isotopic signature in a previous study

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[5]. Average sedimentation rates of 0.27 cm/kyr (09JPC) and 0.23 cm/kyr (14JPC) had been calculated based on the decrease of ^{10}Be/^{9}Be in the core. With the new ^{10}Be half-life of 1.387 Myr [8,9] these values change to 0.29 cm/kyr (09JPC) and 0.25 cm/kyr (14JPC), respectively.

From each core (09JPC, 14JPC) two samples were chosen to be remeasured with the carrier-free method. This serves also as an additional test of the new method in comparison with a conventional determination of the ^{10}Be/^{9}Be ratio using a combination of AMS and ICP-MS.

2.2. Sample processing

The procedure of leaching the seawater-derived isotopic signature of Be from a marine sediment sample for carrier-free ^{10}Be/^{9}Be measurements is adapted from [10]. About 1 g of sediment is first leached in 40 ml acetate buffer (pH 4.66) for 2 h to dissolve the carbonate fraction. After centrifugation and removal of the supernatant the Be fraction is extracted from the sample in a second leaching: During another 2 h of shaking, the seawater-derived ferromanganese coatings are dissolved in 40 ml of 0.05 M hydroxylamine hydrochloride in 15% acetic acid, buffered to pH 4 with NaOH. After centrifugation the supernatant is evaporated and redissolved in ca. 5 ml 6 M HCl. The Fe is separated on a column containing 2 ml BioRad AG1x8 (100–200 mesh). The resin is first rinsed with 10 ml MilliQ water, cleaned with 10 ml 0.3 M HCl and conditioned with 6 ml 6 M HCl. The sample is then loaded onto the column and the Be fraction is collected immediately, the column is further rinsed with another 5 ml 6 M HCl, which are also collected and combined with the Be fraction for further processing.

Afterwards, the material is evaporated and redissolved in ca. 10 ml 0.4 M oxalic acid. Ca in the material may be precipitated in this step. If this is the case, after a centrifugation the supernatant is again evaporated and redissolved in 5–15 ml oxalic acid, else the original solution is processed.

The next purification step is carried out on 1 ml columns of BioRad AG50x8 (200–400 mesh) [11]. The resin is cleaned with 6 ml 5 M HNO_3 and rinsed with 8 ml MilliQ water. Further, the resin is conditioned with 8 ml 0.4 M oxalic acid. The sample is loaded in a volume of 5–15 ml of the same solution. 15 ml of this solution are used to wash the sample down and to remove cations like Fe, Al and Ti. After rinsing with MilliQ water, 6 ml of 0.5 M HNO_3 are used to elute Na. Finally, the Be fraction is collected in 12 ml 1 M HNO_3.

This step is followed by a coprecipitation with 0.5–1 mg Fe, which is cleaned in advance using a BioRad AG1x8 column to avoid any introduction of ^{9}Be in this step. The sample is dried at 80 °C and afterwards oxidized at 650 °C in a crucible for 1–2 h. After mixing with ca. 4 mg Nb powder (325 mesh) the material is pressed into a cleaned Ti cathode-holder.

For the treatment of sediment samples the hydroxylammonium chloride, acetic acid and sodium hydroxide have pro analysi purity, all other chemicals are used at suprapure level.

2.3. Instrumental settings

The carrier-free Be samples are analyzed at the compact TANDY AMS system, which originally had been designed for ^{14}C measurements at low energies [12]. In the meantime, the setup has been modified: By installing a second magnet on the high-energy (HE) side of the spectrometer the background level for ^{10}Be/^{9}Be measurements has been reduced below 10^{-15} [13]. The ^{10}Be/^{9}Be sample ratios are normalized against the in-house standard S1 with a nominal ^{10}Be/^{9}Be ratio of 9.51×10^{-8}. This value is corrected for the new ^{10}Be half-life [8,9] according to [14].

The separation of the ^{10}B interference from ^{10}Be is achieved by the degrader foil method in the setup described in [13]. When selecting the 1+ charge state after the accelerator and the 2+ charge state after the degrader foil the overall transmission from the low energy side to the detector reaches up to 10%. The stability of the setup within a run is illustrated by the results of our standard S1 during three beam times (Fig. 1). Different tunings and slit settings may be required for appropriate ^{10}B suppression and lead to different values for the standards between the runs.

Typical carrier-free samples prepared from 1 g marine sediment show ^{10}Be/^{9}Be ratios above 10^{-9} and develop currents of 0.5–1 nA. Samples are measured for ca. 600 s and give 10–30 ^{10}Be counts per second. The determination of the ^{10}Be/^{9}Be ratio is more sensitive to background from stable ^{9}Be than it is to contamination with ^{10}Be. Therefore, special attention is turned to the preparation of the samples and the measurement of the low currents.

Currents even below 100 pA can be measured reliably in the new setup. The blank level of the current is checked in several ways: Blanks containing only Nb powder are measured to control any cross contamination during the pressing of the sample material into the sputter targets. The Nb blanks usually show only few pA on the HE side. Fully processed blanks are produced starting from the leaching substance and by treating these blanks in the same way and with the same amount of chemicals as used for the normal samples. Thereby, input of ^{9}Be or ^{10}Be by the chemicals used for the processing and cross contamination during sample preparation and in the ion source can be monitored. These blanks typically show ^{9}Be HE currents of 10–15 pA and ^{10}Be rates of less than 2 counts per minute, which corresponds to ^{10}Be/^{9}Be ratios of $1–2 \times 10^{-9}$. Further, targets of the S1 standard with varying Be content are measured to determine a potential current dependence of the ^{10}Be/^{9}Be ratio. A correction for such a dependence may require an HE current offset of up to 15 pA. Usually, this offset correction on the current already explains the currents of the Nb press blanks and in some cases it is even larger than the current of the fully processed blanks. Either the offset correction or the HE current of the fully processed blanks determine the maximum HE current correction. The total HE current correction therefore is less than 15 pA and it has uncertainties lower than 5 pA, which are derived from the standard deviation of the measured and uncorrected currents of the fully processed blank samples.

Burn-in processes in the target normally reduce the ^{9}Be background in all blank samples, which indicates that this background is introduced by a surface contamination of the targets. Therefore, a minimum burn-in time of 10 s is applied prior to the first measurement on the target. The blank corrections to samples with HE currents above 200 pA are negligible. For samples with lower currents the uncorrected ^{10}Be/^{9}Be ratios are all in the 3σ uncertainty of the blank corrected values.

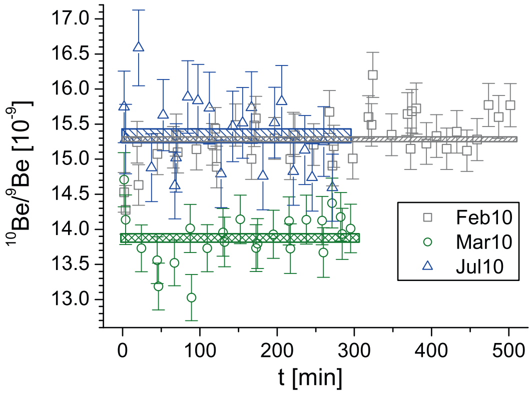

Fig. 1. The results of the standard S1 are presented for the whole time span of the measurement during three different beam times.

3. Results

3.1. Reproducible chemical processing and measurement

From the three ODP sediment samples used in this study the Be content was extracted in several independent preparations (A, B, C in Fig. 2). The three samples show ^{10}Be/^{9}Be ratios in the 10^{-8} range and are characterized by different shapes and colors of the symbols in Fig. 2. The differences in the measurement settings and the chemistry yields are illustrated by the varying currents.

All the results for the ^{10}Be/^{9}Be ratios are consistent for the individual samples. Uncertainties of the single measurements include measurement stability, counting rate, blank correction and the uncertainty of the standard. The overall internal 1σ uncertainty of a measurement on a single target typically is 2–5%. The mean values of the three samples and the according 1σ uncertainties are shown as shaded area in Fig. 2. All but one of the single measurements include the mean value in their 1σ uncertainty, indicating that the single uncertainties of one measurement probably is overestimated. A $\chi^2_{\text{red}} < 1$ is calculated for all three datasets. Obviously, no additional uncertainty is necessary to account for the variations from target to target and from tuning to tuning. In these processings the full reproducibility of the sample preparation and the measurement therefore is smaller than the uncertainty of a single measurement and may amount to a maximum of 2%.

3.2. Sedimentation rates in the Arctic Ocean

Four carrier-free targets were prepared from samples already used to determine the ^{10}Be concentration and the authigenic ^{10}Be/^{9}Be ratio applying the conventional AMS + ICP-MS approach [5]. Our new carrier-free ^{10}Be/^{9}Be values are compared with the corresponding results of this previous study (Table 1, Fig. 3). For the calculation of sedimentation rates only the four samples actually measured in both studies are taken into account.

The currents of parallel processed samples normally do not vary by more than a factor of five. The currents of the four Arctic sediment samples show exceptionally large differences (Table 1), with the samples from the top giving much lower ^{9}Be^{1+} currents than those from the bottom. This might be explained by geochemical variations of the sediments or simply by possible losses during the sample preparation. As we have no elemental data on our samples one can only speculate about variations in the composition: In the chemical processing of the Arctic core samples we observed a green coloration of the resin used for Fe separation instead of the usual yellow coloration. This was particularly pronounced for the core top samples. The coloration of the resin might originate from a special geochemical composition of the sediment, which might influence the Be uptake into the sediment or the efficiency of its chemical separation. However, because of the high ^{10}Be/^{9}Be ratios of the top samples the poor Be output did not limit the counting statistics of these targets but for sample 14JPC-1 the little ^{9}Be^{1+} current caused a large uncertainty.

Table 1
Results for ^{10}Be/^{9}Be ratios (with 1σ uncertainty) and sedimentation rates in Arctic Ocean cores 09JPC and 14JPC of this study and of selected samples determined with the conventional AMS + ICP-MS method [5].

Sample	Depth (m)	Current[1] (pA)	^{10}Be/^{9}Be (10^{-9})	Conv. ^{10}Be/^{9}Be (10^{-9})
09JPC-1	0.02	249 ± 5	23.7 ± 0.6	12.1 ± 0.8
09JPC-11	15.30	718 ± 5	0.65 ± 0.03	0.35 ± 0.04
Sedimentation rate			0.215 cm/kyr	0.212 cm/kyr
14JPC-1	0.60	35 ± 4	10.5 ± 1.3	5.72 ± 0.43
14JPC-8	11.01	1260 ± 5	0.24 ± 0.02	0.12 ± 0.02
Sedimentation rate			0.138 cm/kyr	0.135 cm/kyr

[1] After correction for a stable current offset during the measurement, which is deduced from standard normalization.

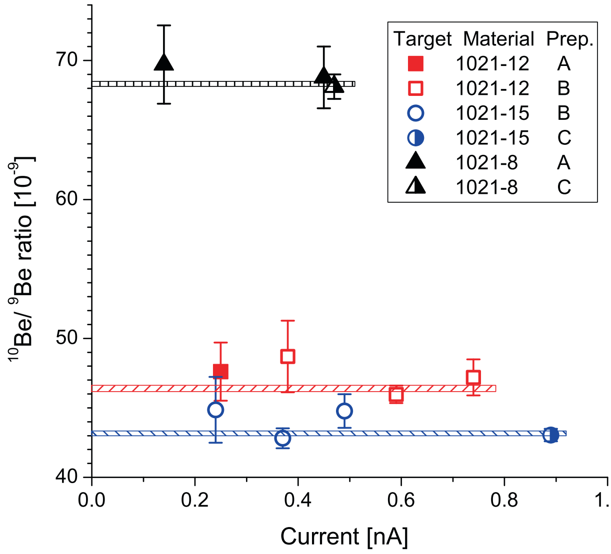

Fig. 2. Sediment samples from different depths at the ODP site 1021 were processed in individual chemical treatments (A, B, C) and their ^{10}Be/^{9}Be ratios were measured in different tunings of the AMS system. These remeasurements of the same target resulted in varying ^{9}Be currents.

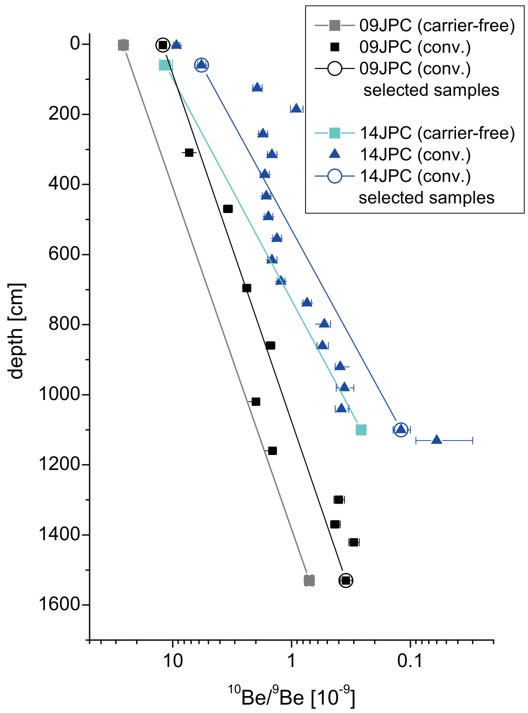

Fig. 3. ^{10}Be/^{9}Be ratios from the cores 09JPC and 14JPC measured with carrier-free and conventional AMS [5] show an exponential decrease with depth.

4. Discussion

The multiple determinations of the ^{10}Be/^{9}Be ratio in the same material prove that the presented preparation of the carrier-free samples gives reproducible results on environmental samples. But further, also the compatibility of the carrier-free values with the results determined using the conventional ^{10}Be/^{9}Be method has to be maintained [4].

4.1. Comparison of the carrier-free and the conventional ^{10}Be/^{9}Be determination

The sedimentation rates determined by [5] could be verified by the carrier-free method to a precision of ±2% if one limits the comparison to the four samples measured in both studies (Table 1). But, if comparing the ^{10}Be/^{9}Be data, an offset between the conventional and the carrier-free determination of this ratio appears. The results obtained with the carrier-free method are systematically higher.

This deviation questions the compatibility of both Be methods. The fully processed blanks show that no contamination with ^{10}Be or ^{9}Be has occurred during the preparation of the carrier-free samples. Accidental leaching of the detritus would cause an excess in ^{9}Be. Therefore, it might only explain the lower ratios derived from the conventional measurement. But in the extraction of Be for the conventional determination the same concentration of the leach solution was used as in our preparation.

Assuming a possible failure during the carrier-free sample preparation, only a contamination with ^{10}Be would explain the higher ratios. However, for all samples a systematic offset by a factor of two would be required, which is unlikely to occur by accidental ^{10}Be input.

During the ICP-MS determination of ^{9}Be, matrix effects might account for the observed offset between the carrier-free and the conventional method. A possible explanation for a too large stable Be concentration measured with ICP-MS might be a progressive dimming of the signal during the different standard addition measurements. But this effect is strongly dependent on the measurement sequence and a substantiated discussion needs further experimental work.

When comparing the ^{10}Be/^{9}Be ratios at the core tops determined with the carrier-free and the conventional method we note that the carrier-free values ($\geqslant 10^{-8}$) match better to values observed in Arctic bottom waters [15]. This is an important control, as the top of the core should represent the recent, seawater-derived ^{10}Be/^{9}Be ratio.

Generally, if a contamination with ^{10}Be can be excluded, a high ^{10}Be/^{9}Be ratio is more plausible: The ^{10}Be is introduced from the atmosphere into the seawater where it mixes with the currently present amount of ^{9}Be. Any non-authigenic ^{9}Be from the sample material, for example released by leaching of the ^{10}Be free detritus, leads to a lower ratio.

In summary, the carrier-free method seems to be more robust concerning contaminations and its results for the authigenic ^{10}Be/^{9}Be ratio in the marine sediments also fit better to seawater values.

4.2. Comparison with other results for sedimentation rates in the Arctic Ocean

The low sedimentation rates in the order of few mm/kyr determined with authigenic ^{10}Be/^{9}Be at the Mendeleev Ridge are questioned by results from dating with biomarkers at a nearby site. A study on racemization rates for two different amino acids in planktonic foraminifera [16] resulted in a sedimentation rate at the site HLY0503-08JPC higher by more than a factor of five. This value matches with the results of an authigenic ^{10}Be/^{9}Be measurement at another Arctic site: A study on the ACEX core from the Lomonosov Ridge in the central Arctic Ocean determined an average sedimentation rate of 1.58 ± 0.1 cm/kyr for the last 11.3 Myr [17, revised for the new ^{10}Be half-life]. This time range was expanded by a second study, finding that 404.8 m of sediment were deposited at a rate of about 1.46 cm/kyr over 27.8 Ma in the last 56.2 Ma [18].

Recently, ^{231}Pa/^{230}Th was used to determine an age model for two other cores from the Mendeleev Ridge [19]. The low sedimentation rates (0.15 cm/kyr) found with ^{231}Pa/^{230}Th agree well with the ^{10}Be/^{9}Be based values of [5] and this study. Generally, sedimentation rates found in the Arctic Ocean range from 0.2 to more than 30 cm/kyr [20].

These strong discrepancies between the different methods or sites are not resolved so far. However, the site HLY0503-8JPC used for racemization dating is situated downslope of the site 09JPC where authigenic ^{10}Be/^{9}Be was applied. Therefore, it has been suggested that enhanced deposition of fine grained material at the slope foot might have led to the higher sedimentation rate determined with the amino acid racemization technique [21]. The strong variations hence may be due to natural causes.

5. Conclusions

The discrepancy for sedimentation rates at the Mendeleev Ridge [5,16] could not be resolved, but our results are in agreement with the minimum estimated sedimentation rate scenario for core 09JPC presented in [5]. We therefore conclude that the new carrier-free method is a useful tool to determine sedimentation rates in the marine environment, comparable to conventional ^{10}Be AMS measurements. Further systematic studies will have to investigate a possible matrix effect on ICP-MS measurements of the stable ^{9}Be, which might explain the observed offset between the two methods for ^{10}Be/^{9}Be ratios in Arctic Ocean sediment samples.

The reliability of the chemical processing for the carrier-free method was tested for a routine operation on marine sediment samples from an ODP core. The processing and the AMS measurement of carrier-free samples in this study have been shown to be stable and reproducible within 2%.

In the future, a study of the Brunhes–Matuyama field reversal will be undertaken with this method.

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