

**Arbeitsgruppe Analytik und kernchemische Grundlagen
Abteilung Nuklearchemie an der Universität zu Köln**

**Working Group of Analytics and Basic Nuclear Chemistry
Division of Nuclear Chemistry at the University of Cologne**

Jahresbericht 2020-21

Annual Report 2020-21

April 2022

Titelbild

Schematische Produktion und Verteilung von ^{10}Be u. ^7Be durch Nass- und Trockenwitterung.
→S. 6;

Coverpage

Schematic production and distribution of ^{10}Be by wet and dry decomposition. ^7Be is formed similarly. →p. 6;

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Laufende Arbeiten / Ongoing Works

Development of an undergraduate lab course experiment from an AMS- ^{41}Ca separation procedure

Marco Michel

Introduction

Of particular interest in the dismantling of the biological shield of nuclear reactors and nuclear power plants is the radionuclide ^{41}Ca , which is formed from the stable ^{40}Ca by neutron activation. Due to its high half-life (approx. $1.02 \cdot 10^5$ y) and high specific activity, it can be used as a reference nuclide during dismantling. ^{41}Ca can be detected based on decay counting methods like liquid scintillation counting (LSC) or based on the mass of the radionuclides using accelerator mass spectrometry (AMS). AMS is preferred since decay-based methods require complete removal of other α - and β -emitters for the measurement. AMS is used to determine the isotope ratio of $^{41}\text{Ca}/^{40}\text{Ca}$, which is currently possible to an accuracy of up to 10^{-16} [1].

Based on the work of Hou, who developed a separation process for ^{41}Ca from concrete for measurements by LSC [2], research of our group was done to modify and simplify it so that an analysis of ^{41}Ca can be achieved via AMS [3] and to determine the yield of this modified separation process [4].

Method

To convert and develop the ^{41}Ca concrete separation process for AMS measurements into an experiment for undergraduate students, the modified process for AMS measurements [3] was simplified in terms by removing time consuming steps. As a result, the analysis by AMS, the investigation of the radionuclide ^{41}Ca and the digestion of a concrete sample were also omitted. In order to demonstrate the efficiency of the separation instead, the concentrations of the natural tracer ^{40}K and the artificially added tracer ^{133}Ba were detected from a concrete-substitute mixture.

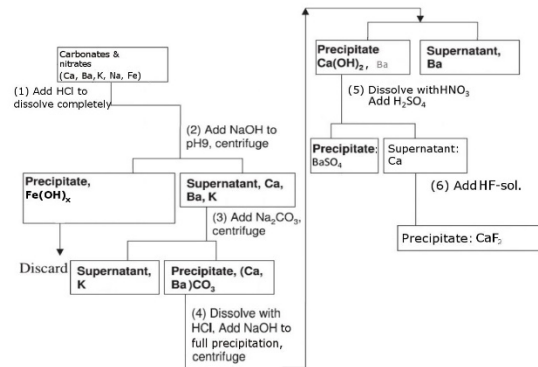


Fig. 1.: Scheme of the separation process used in the experiments.

Results and Discussion

For the separation of ^{133}Ba , no significant activity of ^{133}Ba were detected. Likewise, with the measurement of the ^{40}K , where no significant signals were measured above the background. These results showed that a good efficiency of the simplified separation process for the lab course could be achieved.

Nevertheless, the first tests during the lab course showed that significant activities of both nuclides can be detected in the product. Thus, individual refinements in the separation process could lead to better results.

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Radionuclides of Beryllium in Sewage Sludge

Jessica Kirchhartz

Abstract

In this work, the ratio of the beryllium isotopes ^{10}Be and ^7Be in sewage sludge was investigated. Comparing the long lived ^{10}Be ($t_{1/2} = 1.4 \text{ Ma}$)^[1] and short lived isotope ^7Be ($t_{1/2} = 53 \text{ days}$)^[1], it was the aim to study if there are time depending variations, to deduce possible distribution ways and link the amount of rainfall to the results. First, several samples from the same container of the sewage plant were taken to prove the consistency of this method und then sewage sludge samples from different weeks were tested. For all samples, the activity of ^7Be was measured by gamma spectrometry. Furthermore, all samples were treated with an improved extraction process derived from the bachelor thesis of Jessica Laabs^[2]. Now, multiple samples can be finished in one day, with a yield of $90 \pm 12 \%$ and no detectable ^7Be in the residue. The purification process through column chromatography however revealed difficulties due to overcharging the resin. After adjusting the method, the ^{10}Be amount of two samples could be measured with AMS and gave a $^{10}\text{Be}/^7\text{Be}$ ratio of 30 ± 4 , but to answer the raised questions, more samples have to be processed.

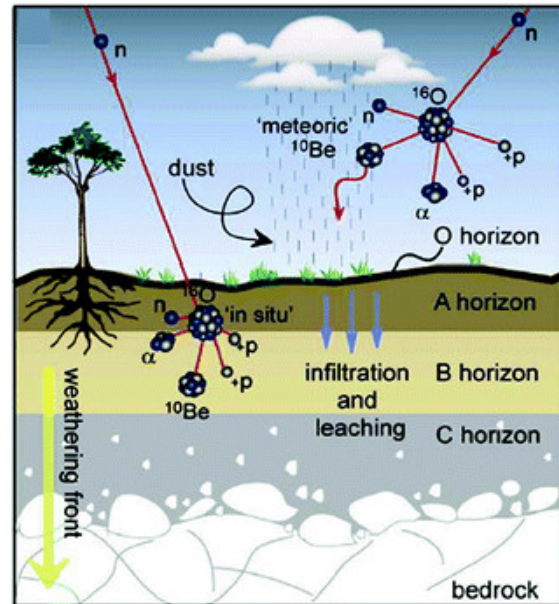


Fig. 1.: Schematic production and distribution of ^{10}Be by wet and dry decomposition. ^7Be is formed similarly.^[1]

References

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Non-invasive analysis of fire gilded layers

Raphael Margreiter

The initial observation leading into this work was that fire gilded silver [1] objects from the 1st century AD in the Antikensammlung Berlin showed low but clearly enriched Hg contents between 0.5% and 5% [2]. The previously accepted minimum limit for Hg in fire gilded objects was 5%, leaving the Hg contents in those 1st century samples unexplained [3]. This led to two main questions that this work tried to answer.

1. Does the 5% minimum need to be abolished? Can we reliably reproduce such low Hg contents in our own fire gilded samples, and if so, how?
2. Can we find another method for the identification of fire gilding except for the pure presence of mercury, based on other characteristics of fire gilded samples?

In all our samples, a Hg content of even 10 % is rarely undercut, there are very few single measurements that undercut the accepted 5% minimum limit, but those were part of an inconclusive measurement series and not robust enough to challenge the accepted limit. Additionally, even visually destroyed artificial aging samples showed Hg contents above the limit. Generally speaking, our measurements further substantiate the accepted limit.

In addition, we found a Hg enrichment at the interface of fire gilded Ag substrates that forms quickly after the amalgam application due to diffusion of Ag and Hg.

There is no reason to believe that this characteristic vanishes over time. Furthermore, with the Hg enrichment at the interface, superficial influences of the sample surrounding such as leaching in the soil will not significantly reduce the measured Hg content.

From all this, the elevated Hg content in the 1st century samples of the

Antikensammlung Berlin are probably not explained by fire gilding. The concentration of these samples on a single century, on the other hand, suggests that they are probably not explained with flawed XRF measurements.

It might be, that our artificial aging measurements were not sufficient as they did not take the influences of the environment, e.g., soil, into account, potentially leading to a depletion of Hg in the sample.

On the other hand, the Hg contents might be explained by the introduction of Hg into the workspace of ancient goldsmiths during the 1st century. Hg vapor could diffuse into separately stored gold, which would be used for other types of gilding.

Regarding identification beyond the 5% criteria, we found a clear Hg enrichment at the interface of fire gilded Ag substrates. This characteristic could be identified with

destruction-free methods by combining techniques with different depth information.

Some preliminary tests were performed during this thesis, such as PIXE measurements with yet to be understood results and a rough series of low angle measurements with inconclusive results, probably due to suboptimal surface conditions.

No characteristic of fire gilded Cu substrates could be found.

References

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Studies on Rhenium and Technetium compounds with SO₃

Stefan Sutorius

In this work, the chemistry of the rhenium sulphate system was expanded. The compound $(C_5NH_6)_3[Re_2O_3(S_2O_7)_4]$ was synthesized by unusual synthesis methods and characterized by means of single crystal diffractometry. For the first time, pure SO₃ was used, acting as both solvent and reactant. The possibility of generating SO₃ without using an apparatus as described in Chapter 3.2 was developed. Different synthesis routes were carried out to find the most effective synthesis method. In addition, the amounts of the reagents used were broken down so that synthesis could be carried out in the 2-4 mg range.

This made it possible to carry out analogous syntheses with technetium-containing reactants. Here, however, no single crystals could be generated. However, a change in the color of the technetium was observed, confirming the existence of a reaction. The colorless

ammonium pertechnetate educt turned into a green solution after the synthesis.

In future experiments, it should be attempted to optimize and refine the synthesis conditions of the reaction with the ammonium pertechnetate so that the growth of single crystals can take place. If this is not successful, spectroscopic methods such as infrared spectroscopy or Raman spectroscopy can be used to examine the green solution obtained. This would allow the environment of the Tc particles to be further characterized.

Further investigations on the $(C_5NH_6)_3[Re_2O_3(S_2O_7)_4]$ should also be carried out. The same spectroscopic investigations as for the technetium experiments, i.e. IR and Raman spectroscopy, can clarify further properties. In addition, investigations such as XPS can be carried out to find out whether $(C_5NH_6)_3[Re_2O_3(S_2O_7)_4]$ is really a mixed valent compound, as is already suspected.

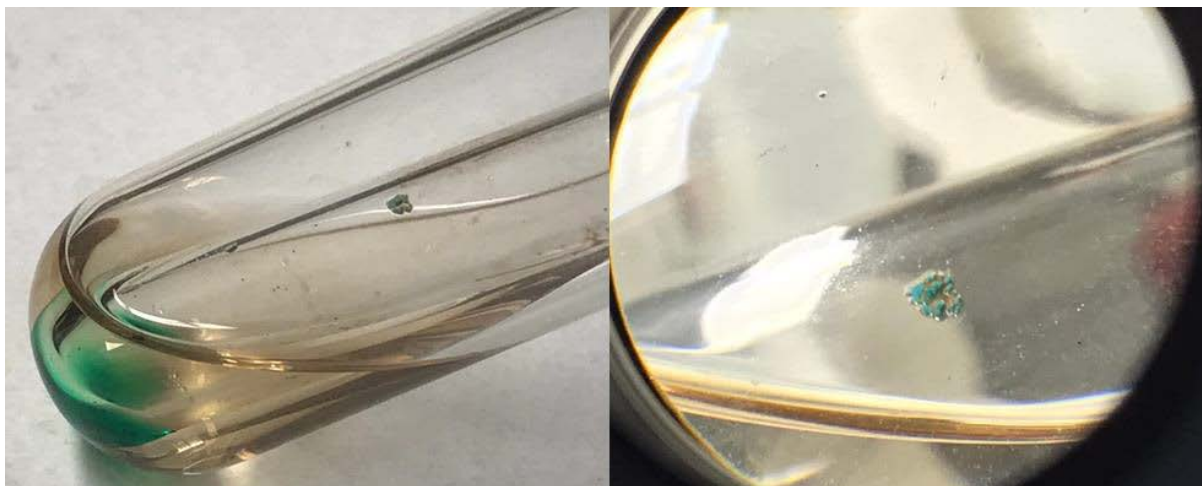


Figure 17: (Left) Images of the green technetium solution. (Right) Possible blue crystalline solid.

Isobar separation limits of Accelerator Mass Spectrometry and comprehensive approach by Ion Laser Photodetachment and Electron Transfer Reactions

M. Schiffer, O. Marchhart, S. Herb, M. Martschini, R. Golser,

E. Strub, A. Dewald

¹Accelerator Mass Spectrometry has demonstrated its strength in the detection of rare radionuclides in the attogram (10⁻¹⁸ g) range as a result of combining efficient ion beam production and filtering. Such low levels of detection (LOD) are a result of dedicated sample preparation, anion selection, molecule dissociation and isobar suppression. The overall limiting factor for the detection of single nuclei by direct atom counting is the background of ubiquitous stable isobars. The isobar suppression is done generally by discriminating isotope of interest and isobars via their respective energy loss, since they have a difference in their proton number. In particular, the differentiation can be done by passive energy loss (stopping power) measurement, like gas-filled magnets or absorber foils, or by active methods, for example by a gas ionization detector. Furthermore, the suppression with a gas-filled magnet is enhanced due to the difference in the mean charge states. In any case, a high kinetic energy is needed if the relative proton number differences are small. Only then, the deviation of the stopping powers is big enough to overcome statistical variances and to allow a reliable particle identification. In the case of highly abundant stable isobars, like ⁶⁰Fe/⁶⁰Ni and ⁵³Mn/⁵³Cr, this kinetic energy can only be reached with big tandem accelerators with terminal voltages in the range of 10 MV. For this reason, the medium mass range (e.g. ⁹³Zr, ⁹⁹Tc) represents the limit for conventional isobar separation techniques. For heavier radionuclides with lower relative proton number difference to the stable isobar it is difficult to reach a sufficient LOD for radionuclides in natural concentrations, which are of great importance for societal questions like ⁹⁹Tc

and ¹³⁵Cs or for astrophysical research like ¹⁸²Hf. However, radionuclides whose isobars do not form negative ions (e.g. ⁵⁵Fe, ¹²⁹I), or which have no stable isobar (e.g. ²³⁶U, ²⁴⁴Pu) are nevertheless precisely measurable with AMS and the highest sensitivities are reached for such radionuclides. An advanced approach for the suppression of highly abundant stable isobars is the selective laser photodetachment of isobar anions. The difference in the electron affinity of the isobars allows to neutralize anions of a specific element by laser irradiation with a photon energy above their EA, while leaving the ion of interest with higher EA unaffected. This technique of Ion Laser InterAction Mass Spectrometry (ILIAMS) has demonstrated an extraordinarily high isobar suppression capability for a variety of radionuclides, like ²⁶Al, ³⁶Cl, ⁹⁰Sr, and ¹³⁵Cs [1]. For sufficient isobar suppression, long interaction times on the order of ms are needed, and the ion beam must be decelerated after the extraction from the ion source in a gas-filled radio-frequency quadrupole (RFQ) ion cooler. The admixture of reactive O₂ gas to the helium buffer gas has revealed an even higher isobar suppression for ⁹⁰Sr, as a consequence of electron transfer reactions and ion-molecule transfer reactions. Gas-filled RFQs are not commercially available, and their development is in the focus of ongoing research with exotic nuclei. A crucial point is the efficient deceleration and trapping of ion beams with high beam emittance like heavy molecular anions, e.g. ⁹⁰SrF³⁻ or ¹⁸²HfF⁻. Sophisticated ion optical injection structures, like an elliptical injection electrode, comparable to ISOLTRAP [2], allow to slow down the ions far away from the central region where the

¹ This text is the abstract submitted to the IAEA Technical Meeting on Developments in Non-

buffer gas is leaking out of the RFQ through the central aperture. Despite of modern machining techniques for complex mechanical structures, technical challenges of aligning had to be solved for a reliable operation. To guide the thermalized anions through the gas- filled RFQ, a gradually changing potential in longitudinal direction is of particular importance and can be created by diagonally split cylindrical electrodes, which are capacitively coupled to a core rod that is carrying the RF signal. This advanced design simplifies the complex structure of diagonally split hyperbolic RF electrodes of the BECOLA setup [3]. The expected benefits of such an arrangement are the low required DC voltages and additionally the low multipole expansion coefficients.

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Radiological characterization for nuclear decommissioning with accelerator mass spectrometry

E. Strub, R. Margreiter, M. Michel, D. Elchine, A. Stolz, T. Pabst, G. Hackenberg, S. Heinze, S. Herb, M. Schiffer, C. Müller-Gatermann, A. Dewald, R. Spanier, M. Dewald, B. Dittmann, K. Eberhardt

²In the field of nuclear waste management, the quantification of a variety of nuclides is important for the radiological characterization of a material. Commonly used techniques for detection rely on the gamma decay of those nuclides. Since there are several nuclides like e.g., ³H, ³⁶Cl, ⁴¹Ca or ⁹⁰Sr, which undergo beta decay and emit no or only a weak gamma particle, other techniques have to be used. Common ones rely on the beta decay and become time and cost intensive with increasing half-life of the measured nuclide. Hence, accelerator mass spectrometry (AMS) can be a promising alternative in detecting those nuclides since it does not rely on the decay but measures and counts the atoms directly. Commonly used reference nuclides in nuclear waste management are, e.g., ⁶⁰Co or ¹⁵²Eu because they can be easily quantified by gamma ray spectrometry. In case of ⁶⁰Co the relatively short half-life of 5.3 a might be a major disadvantage as decommissioning works might take place on a longer timescale. Thus, it is not very well suited if longer storage and control is needed. Another disadvantage in the case of reactor concrete is that ⁶⁰Co and ¹⁵²Eu are generated by neutron irradiation of ⁵⁹Co or ¹⁵¹Eu, respectively, which both are contained only as trace elements with an unknown level of homogeneity. A research project therefore investigated the suitability of ⁴¹Ca as a reference isotope for the characterization of reactor concrete, e.g., originating from the bio-shield of a nuclear power plant. For this purpose, several samples of concrete material were irradiated with thermal neutrons with different doses at the Mainz TRIGA reactor. The irradiated samples were then measured by gamma spectrometry and

⁴¹Ca was chemically extracted as CaF₂ to produce a sputter target for the AMS measurement. Subsequently, the ⁴¹Ca concentration was successfully determined at the 6 MV tandem AMS set-up CologneAMS at the University of Cologne

Activated graphite, e.g., from graphite moderated reactors, contains besides ¹⁴C several radioactive nuclides with pure beta decay like ³⁶Cl or ³H. The institute for nuclear physics in Cologne is aiming for a system which enables automated measurements using the AMS technique with gaseous samples, for ¹⁴C, ³⁶Cl, and ³H. The planned system will provide a high sample throughput as well as the possibility of sample dilution in cases of high activity. Therefore, a new gas-interface was built, which uses two syringes for the transport of the sample gas into the ion source and a separate reservoir for blank gas which can be used for the dilution. ⁹⁰Sr is produced by nuclear fission and is a prominent nuclide in nuclear waste and fallout. Currently AMS is capable of measuring ⁹⁰Sr/Sr ratios below 10⁻¹², which corresponds to an activity of less than 4 mBq. Therefore, ⁹⁰Sr is extracted as SrF₃ in the ion source. The limiting factors for lower ratios are the background of the isobar ⁹⁰Zr and the weak ion source output. Since the suppression of the isobar ⁹⁰Zr demands a high accelerator energy, CologneAMS is developing the measurement of ⁹⁰Sr at the 10-MV tandem accelerator at the institute for nuclear physics. The target preparation is performed at the department for nuclear chemistry, Cologne. The recent status and development will be shown.

² This text is an abstract of a contribution submitted to the IAEA Technical Meeting on Developments in Non-Radiocarbon Accelerator

An advanced radio-frequency quadrupole ion cooler for Accelerator Mass Spectrometry

M. Schiffer, O. Marchhart, S. Herb, M. Martschini, R. Golser,

E. Strub, T. Dunai., A. Dewald

³Ion Laser InterAction Mass Spectrometry (ILIAMS) has demonstrated an extraordinarily high isobar suppression capability for a variety of radionuclides which are important for accelerator mass spectrometry. It uses selective laser photodetachment of decelerated ion beams in a gas-filled radio-frequency quadrupole (RFQ) cooler for the suppression of interfering isobars [1]. Furthermore, the admixture of O₂ gas (≈3%) to the helium buffer gas has revealed an even higher isobar suppression, larger than 10⁵ in the case of ⁹⁰Sr/⁹⁰Zr, at the Vienna Environmental Research Accelerator (VERA), without the use of a laser. Therefore, we started to develop a RFQ cooler designed for the deceleration and trapping of ion beams with high beam emittance like heavy molecular anions, e.g. ⁹⁰SrF₃. The system will be finally used at the Cologne 6 MV accelerator after commissioning and test measurements at VERA. The ion cooler will use gas reactions with the option of adding a laser in a later phase. The new RFQ design intends to solve technical challenges by a self-aligned structure with the possibility of readjustment without breaking the vacuum. The vacuum chamber of the RFQ can be opened at the top for easy maintenance and for changes in the experimental setup. This allows changes of the quadrupole itself, like the installation of higher-order multipole segments at the entrance of the radio-frequency device providing higher acceptance for divergent ion beams. Based on the design of the injection unit of ISOLTRAP [2] an elliptical injection electrode was developed that should increase the theoretical transmission in comparison to ring or conical electrodes. It will also allow to slow down the ions far

away from the central region where the buffer gas is leaking out of the RFQ trough the central aperture. A new and easy to manufacture guide field assembly was developed to reduce the electric multipole order, which is naturally induced by inclined DC electrodes. For this purpose, diagonally split cylindrical electrodes are capacitively coupled to a core rod that is carrying the RF signal. Consequently, only low DC voltages are needed to create a gradually changing potential in longitudinal direction. This design is a simplification of the diagonally split hyperbolic RF electrode assembly of BECOLA [3]. In this contribution, we will compare different guiding field structures by the calculation of multipole expansion coefficients. Additionally, a remote-controlled radio-frequency resonance tuning and impedance matching system for heavy radionuclide applications will be presented, which is able to drive the RFQ with frequencies in the range of 1 MHz with RF peak-to-peak voltages of 250 V.

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³ This text is an abstract of a contribution submitted to the AMS-15 conference

AMS measurements of C-14, Cl-36, and Pu-239/240 samples taken in a small facility

M. Michel, A. Filby, S. Heinze, R. Margreiter, E. Strub

⁴In Germany, the release of nuclear facilities from regulatory control is commonly addressed by demonstrating that possible contaminations with radionuclides are below the clearance values as given in the German Radiation Protection Ordinance. For certain radionuclides, those clearance levels are very low, e.g. 1 Bq/g for the unrestricted release of C-14. Such a limit might be a challenge for conventional decay counting techniques, especially regarding weak beta emitters like C-14 and Cl-36. Also, Pu-240 poses a challenge because it can practically not be distinguished from Pu-239 by alpha spectrometry since the respective alpha energies are very similar. However, for the release of a bigger nuclear facility from regulatory control, this poses no problem: it is good practice to define nuclide vectors, i.e. assuming a fixed composition of different radionuclides. Typically, these nuclide vectors contain some easily detectable nuclides like Co-60 as reference nuclides. The quantification of the radionuclides is then derived from the measurement results of these reference nuclides. Contrary, in the case of so-called small facilities, like radiochemistry laboratories, the operational history might be unclear and/or a broad range of different radionuclides might have been utilized. In such cases, it might also be challenging to derive a common nuclide vector and it might be necessary to quantify radionuclides that are difficult to measure. Also in such cases, AMS (accelerator mass spectrometry) is probably not the obvious choice for an analytical technique. However, this work aimed to demonstrate that AMS is able to provide valuable information for the release of small facilities from regulatory control. For this case study, a radiochemistry laboratory in Cologne, Germany was chosen. It was known that

C-14, Cl-36 as well as Pu-240 had been handled openly in this lab. There were no apparent contaminations, i.e. no alpha/beta contaminations that could be detected using a hand-held radiation monitor. In total, 10 concrete samples were taken out of the walls and the floors of the facility and processed for the subsequent AMS measurements.

⁴ This text is an abstract of a contribution submitted to the AMS-15 conference

Characterisation of Reactor Graphite with Ion Beam Techniques

M. Gwozdz, T.-F. Pabst, M. Steffan, G. Hackenberg, K. Eberhardt, S. Heinze, S. Herb, M. Schiffer, A. Stolz, E. Strub, A. Dewald

⁵Activated graphite, e.g. from graphite moderated reactors contains several radioactive isotopes like ¹⁴C, ³⁶Cl, or ³H. For the final disposal of such material a quantitative characterization is demanded. We are aiming for a system which enables automated measurements using the AMS technique with gaseous samples, especially for the isotopes ¹⁴C, ³⁶Cl, and ³H. The aimed system should provide a high sample throughput as well as the possibility of sample dilution in cases of high activity. Therefore, for ¹⁴C, a new gas-interface was built which uses two syringes for the transport of the sample gas into the ion source and separate reservoir for blank gas which can be used for the dilution. An advantage over the already established procedure using Liquid Scintillation Counting (LSC) is that the setup at the CologneAMS does not need elaborate chemical sample preparation. Additionally the LSC method is less sensitive with 2·10E-4 Bq/g whereas the AMS technique can be used down to 3·10E-9 Bq/g [1]. For the measurement of the tritium concentration, we expanded our ion source test bench. A 100 kV accelerator stage with a carbon stripper foil in the centre was installed along with an additional 90° analysing magnet and a silicon detector. This will allow efficient and accurate characterisation of the activity in reactor graphite material, which is foreseen to be stored in repositories like the e.g. mine Konrad where activity limits have to be considered. In this contribution we will present the layout of our systems as well as the stage of realization. The project was supported by BMBF under contract number 15S9410B.

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⁵ This text is an abstract of a contribution submitted to the AMS-15 conference

Proof of suitability of Ca-41 as a reference isotope for the characterization of reactor concrete

R. Spanier, M. Dewald, B. Dittmann, S. Heinze, M. Schiffer, S. Herb, C. Müller-Gatermann, G. Hackenberg, A. Stolz, A. Dewald, R. Margreiter, E. Strub, M. Michel, K. Eberhardt

In the field of nuclear waste management, reference nuclides are important for the radiological characterization of the radioactive material. Commonly used reference nuclides are e.g. Co-60 or Eu-152 because they can be easily quantified by gamma ray spectrometry. In case of Co-60 the relatively short half-life of 5.3 a might be a major disadvantage as decommissioning works might take place on a longer timescale. Thus, it is not very well suited if longer storage and control is needed. Another disadvantage in the case of reactor concrete is that they are generated by neutron irradiation of Co-59 or Eu-151, respectively, which both are contained only as trace elements with an unknown level of homogeneity. This research project therefore investigated the suitability of Ca-41 as a reference isotope for the characterisation of reactor concrete, e.g. originating from the bio-shield of a nuclear power station. For this purpose, several samples of concrete material were irradiated with thermal neutrons with different doses at the Mainz TRIGA reactor. The irradiated samples were then measured by gamma spectroscopy and the Ca-41 was chemically extracted as CaF to produce a sputter target for the AMS measurement. Subsequently the Ca-41 concentration was determined at the 6MV TANDETRON AMS set-up, "CologneAMS", of the University of Cologne. We will report on the results with respect to sensitivity and precision obtained so far.

⁶ This text is an abstract of a contribution submitted to the AMS-15 conference

Quantification of C-14 in artificially activated reactor graphite via AMS

R. Margreiter, M. Dewald, M. Gwozdz, S. Heinze, A. Dewald, K. Eberhardt, E. Strub

⁷There are currently 250.000 Mg activated reactor graphite around the globe without a well-defined disposal path [1]. A relevant subgroup of this material is the roughly 1000 Mg reactor graphite in Germany, largely from the prototype reactors AVR (Jülich) and THTR-300 (Hamm-Uentrop). The disposal of this material is as of now still an unresolved challenge. The still ongoing research presented here tries to address some of the difficulties and to open a possible path toward an orderly disposal procedure, mainly by investigating the possible application of the CologneAMS system for precise quantification of C-14 in reactor graphite.

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⁷ This text is an abstract of a contribution submitted to the AMS-15 conference

Weiterentwicklung der Messung von Strontium-90 mittels LSC

Dominik Elchine

Zusammenfassung

⁸In dieser Arbeit wurde sowohl das Region of interest (ROI) im LSC β -Spektrum von sich im säkularen Gleichgewicht befindlichen ^{90}Sr und ^{90}Y als auch das ROI vom β -Spektrum mit dem Gleichgewichtsfall $^{90}\text{Sr}/^{90}\text{Y}$ auf das Figure of Merit (FOM) untersucht. Da schmale Kanalbereiche für einen hohen FOM-Wert genutzt wurden, wurden Multiplikationsfaktoren berechnet, um die Gesamtzählrate zu bestimmen. Die Basis für die Bestimmung der Multiplikationsfaktoren bilden 92 individuell ausgewertete β -Spektren mit einer Messzeit von 14400 s. Die jeweiligen K-Faktoren für jedes Spektrum wurden arithmetisch gemittelt. Die Multiplikationsfaktoren der verschiedenen ROI und deren arithmetisch gemittelter Fehler wurden verglichen, um eine Aussage über ihre Konsistenz und Genauigkeit treffen zu können.

Im Anschluss wurden die Detektionslimits der aus der FOM Analyse ausgewählten ROI bestimmt, die dann mit dem ROI aus der Referenzliteratur verglichen wurden. Dafür wurden zwei Stammlösungen mit ^{90}Sr und ^{90}Y im säkularen Gleichgewicht angesetzt. Die Stammlösungen wurden quantifiziert. Die Nachweisgrenzen wurden ermittelt, tabellarisch aufgezeigt und mit den Werten der aus *Weler et al. (2017)* stammenden Regionen verglichen. Für Cerenkov Messungen von ^{90}Y wurde ebenfalls eine Quantifizierung einer verdünnten Stammlösung durchgeführt, welche auf die Nachweisgrenze zweier ROI untersucht wurde. Auf Grundlage der erstellten Quantifizierung der Stammlösungen wurden AMS-Standards vom ^{90}Sr für das AMS Cologne hergestellt. Mithilfe des optimierten Bereiches für Cerenkov Messungen wurden

Klärschlammproben auf den ^{90}Y Gehalt untersucht, um Rückschlüsse daraus ziehen zu können wie viel ^{90}Sr in der Klärschlammprobe enthalten ist. Darüber hinaus wurde ^{90}Y vom ^{90}Sr getrennt. Das reine ^{90}Sr wurde dann über 3 Wochen gemessen, damit klar wurde nach welcher Zeit sich das säkulare Gleichgewicht nachbildet.

⁸ This text is identical with the abstract of the corresponding thesis.

Radiochemische Analyse von bestrahltem Reaktorgraphit

Florian Putkamer

Zusammenfassung

⁹Im Rahmen dieser Arbeit wurden bestrahlte Reaktorgraphit-Proben auf die in der Graphit-Matrix eingeschlossenen langlebigen Radionuklide untersucht. Die drei Proben wurden bereits einige Zeit vor dieser Arbeit am TRIGA-Reaktor in Mainz im Rahmen eines Modellversuchs mit Neutronen bestrahlt.

Durch unterschiedliche radiochemische Analysemethoden wurden die langlebigen aktivierten Nuklide bestimmt. Für die Messung der charakteristischen Röntgenstrahlung und die Messung von β -Strahlern am LSC (Liquid Scintillation Counting) mussten die Proben chemisch aufgeschlossen werden. Hierbei wurden unterschiedliche Aufschlussmethoden eingesetzt. Es zeigte sich hierbei, dass sich für die anschließende Analyse von flüchtigen Bestandteilen am besten eine Pyrolyse mit dem anschließenden Auswaschen der Radionuklide eignet. Für den Aufschluss von schwer löslichen Bestandteilen konnte mit Hilfe einer Hochdruckveraschung ein guter Reaktionsumsatz erzielt werden. Am LSC konnten Tritium, Kohlenstoff-14 und Chlor-36 nachgewiesen werden.

Zusätzlich konnten durch Langzeitmessung der bestrahlten Proben am γ -Detektor weitere Radionuklide nachgewiesen werden. Mit Röntgen- und γ -Spektrometrie konnten vor allem Spurenelemente des d-Blocks sowie Kalium-40 nachgewiesen werden.

⁹ This text is identical with the abstract of the corresponding thesis.

Entwicklung einer thermochromatographischen Trennmethode für Mangan aus Chrom

Salim Gülez

Zusammenfassung

¹⁰In dieser Arbeit wird ein thermochromatographisches Trennsystem zur Trennung trägerfreier Mengen des Mangans entwickelt. Dazu wurde ein thermochromatographisches Trennsystem aufgebaut und untersucht für die Trennung der 3d bis 5d Elemente der Gruppe 6 und 7.

Es wurde ein Thermochromatograph basierend auf Voruntersuchungen in der Literatur aufgebaut und weiterentwickelt. Die Thermochromatographie wurde mit Trennungen der Nuklide ⁹⁹Mo, ^{99m}Tc, ⁵⁶Mn, ⁵¹Cr, ¹⁸⁶Re, ¹⁸⁸Re und ¹⁸⁷W durchgeführt.

Es konnten erfolgreich die 3d- und 5d-Elemente getrennt werden. Die Trennung der 4d-Elemente war aufgrund der Probenbeschaffenheit schwer nachzuvollziehen.

¹⁰ This text is identical with the abstract of the corresponding thesis.

Charakterisierung des MOFs UoC-3 als Kationenaustauscher für Cäsium

Majied Sadegh Zadeh Farid

Zusammenfassung

¹¹Das Ziel dieser Arbeit ist die Charakterisierung der metallorganischen Gerüstverbindung (metal organic Framework, MOF), $[(\text{CH}_3)_2\text{NH}_2][\text{UO}_2(3\text{F-BTB})] \cdot x \text{ DMF}$ UoC-3 als Kationenaustauscher für Cäsium-137 (¹³⁷Cs). Das MOF UoC-3 besteht aus einem Uranyl-Knotenpunkt und dem organischen Linker 1,3,5-Trifluoro-2,4,6-tris(4-carboxyphenyl)benzol ($\text{H}_3\text{-3F-BTB}$) sowie dem organischen Kation $[(\text{CH}_3)_2\text{NH}_2]^+$, welches selektiv mit Cs^+ ausgetauscht wird. Um das MOF mittels Solvothermalsynthese zu synthetisieren, werden die Edukte mit einem Lösungsmittel bei erhöhter Temperatur in einem Autoklaven umgesetzt.

In dieser Arbeit wurde das MOF nach einer bestehenden Vorschrift in einer ausreichenden Menge für Ionenaustauschexperimente synthetisiert. Anschließend wurde die Aufnahme von Cs^+ unter Betrachtung verschiedener Zeitintervalle, sowie die Kapazität und die Selektivität des MOFs in Anwesenheit von Natrium und Kalium bestimmt. Dafür wurde trägerfreies ¹³⁷Cs eingesetzt. Ein weiteres Ziel dieser Arbeit war die Untersuchung der Regenerationsfähigkeit des MOFs, um eine mehrfache Nutzung zu ermöglichen. Die Versuche wurden mit einem Koordinationspolymer (Coordination polymer, CP) $[(\text{CH}_3)_2\text{NH}_2][\text{UO}_2(\text{BTC})]$ wiederholt und mit den Messergebnissen des MOFs verglichen. Das CP wurde mit den organischen Kationen $[(\text{CH}_3)_2\text{NH}_2]^+$ und $[(\text{C}_4\text{H}_9)_4\text{N}]^+$ sowie unterschiedlich funktionalisierten Linkern synthetisiert.

Es wurde eine Cäsiumaufnahme sowie eine gewisse Selektivität in Anwesenheit von konkurrierenden Alkalimetallen (K^+ und Na^+) beobachtet. Eine Regeneration des

MOFs zur erneuten Nutzung erwies sich als schwierig. Die besten Ergebnisse für die Cäsiumaufnahme wurden mit einem unfluorierten CP erhalten.

¹¹ This text is identical with the abstract of the corresponding thesis.

Ionenaustauschereigenschaften von MOFs und Koordinationspolymeren

Melisa Maslo

Zusammenfassung

¹²Das Ziel der vorliegenden Bachelorarbeit war eine nähere Untersuchung der Ionenaustauschfähigkeiten des MOFs (Metall Organic Framework) UoC-3 [(CH₃)₂NH₂][UO₂(3F-BTB)] und der CPs (engl. *Coordination Polymer*) [(CH₃)₂NH₂][UO₂(mF-BTC)] bzw. [(CH₃)₂NH₂][UO₂(BTC)] in Gegenwart von ¹³⁷Cs.^[1] Aspekte, die dabei untersucht werden, sind Eigenschaften wie die Regenerationsfähigkeiten sowie die Affinität gegenüber ¹³⁷Cs.^[1] Vorhergehende Untersuchungen deuteten darauf hin, dass eine Adsorption des ¹³⁷Cs sowohl bei den CPs als auch den MOFs zu beobachten ist.^[1] Bei Betrachtung der Regenerationsfähigkeit der beiden vorliegenden Systeme konnte eine geringe Regeneration festgestellt werden.^[1]

Des Weiteren wurde der Einfluss des pH-Wertes auf den Verteilungskoeffizienten K_d untersucht. Hierfür wurden Langzeituntersuchungen mit verschiedenen alkalischen Lösungen durchgeführt. Neben der Adsorptionsfähigkeit wurde auch die Ionenselektivität in Bezug auf das ¹³⁷Cs in Gegenwart anderer Kationen untersucht. Diese Untersuchungen sind relevant, da die MOFs und CPs zur Aufreinigung von kontaminiertem Wasser dienen könnten. Ein Beispiel wäre das verwendete Kühlwasser bei der Katastrophe von Fukushima.^[2] Bei dem verwendeten Kühlwasser handelte es sich um Meerwasser, weshalb es eine hohe Konzentration an Natrium-Kationen enthält sowie zu einem geringeren Anteil Kalium-Kationen.^{[2][3]} Darüber hinaus wurde die Kapazität des MOFs mit Hilfe einer Verdünnungsreihe von CsBr bestimmt.^[1] Ein weiterer Aspekt war eine

Vorkonditionierung des MOFs UoC-3 mit Lithiumhydroxid.

Quellen

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¹² This text is identical with the abstract of the corresponding thesis.

XRF-Messungen an Münzmetallen

Sven Lemke

Zusammenfassung

¹³In der Numismatik können nicht nur Daten wie Alter, Gewicht und die Dichte einer antiken Münze von Relevanz sein, auch deren chemische Zusammensetzung kann von großem Interesse sein, um beispielsweise antike Herstellungsprozesse bzw. Herstellungspraktiken oder Provenienzen zu analysieren. Um die Zusammensetzung zu ermitteln, werden antike Gegenstände nach Möglichkeit mit zerstörungsfreien Methoden, wie etwa Röntgenfluoreszenzspektroskopie, analysiert.

Um die automatischen Analysenergebnisse von Messungen an Münzen zu überprüfen und ggf. kalibrieren zu können, wurden in dieser Arbeit 42 verschiedene Bleibronzelegierungen mit unterschiedlichen Anteilen an Kupfer, Zinn und Blei hergestellt und mit dem XRF-Spektrometer vermessen. Die erhaltenen Daten zeigten, dass der Zinngehalt der Proben sehr genau der getätigten Einwaage entspricht. Der Bleigehalt wurde hingegen systematisch unterschätzt, wohingegen der Kupferanteil überschätzt wurde. Dies kann durch Matrixeffekte zwischen den Blei- und Kupferatomen erklärt werden. Aus den Messdaten wurde ein Korrekturfaktor für den Bleianteil ermittelt, der Kupferanteil konnte komplementär zur Differenz des Bleianteils korrigiert werden. Außerdem wurde eine Auswahl antiker Münzen aus der Sammlung der Universität zu Köln nach ihren vorerfassten magnetischen Eigenschaften getroffen und erstmals mit einem XRF-Spektrometer vermessen und die Daten wie oben beschrieben korrigiert, um die wahren Zusammensetzungen präzise zu ermitteln. Zudem wurden die magnetischen Suszeptibilitäten einiger Zusammensetzungen bestimmt und mit

Münzen, welche eine ähnliche Zusammensetzung aufweisen, verglichen. Dabei konnte jedoch keine einfache Interpretation der magnetischen Daten im Zusammenhang mit ihrer chemischen Zusammensetzung abgeleitet werden.

¹³ This text is identical with the abstract of the corresponding thesis.

3D-Druck für das Chemielabor

Sebastian von Döllen, Salim Gülez, Erik Strub

Bei dem eingesetzten 3D-Drucker handelt es sich um den Ender 3 Pro der Firma Creality. Im Preis-Leistungs-Verhältnis ist dieser 3D-Drucker quasi all seinen Konkurrenten überlegen, was im Wesentlichen darin begründet liegt, dass es sich um ein Open Source Projekt handelt, was sowohl die Software wie auch die Hardware betrifft.

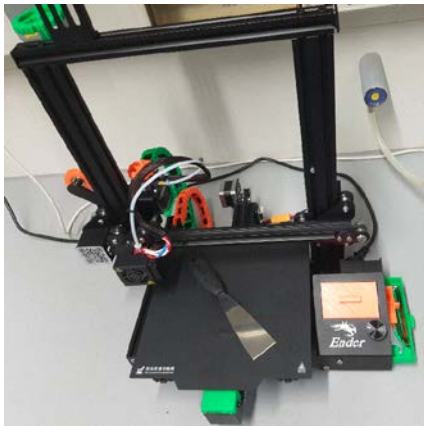


Abbildung 1: 3D Drucker Creality Ender 3 Pro.

Betreffend aktuelle Forschungsprojekte, hat sich die Möglichkeit des 3D-Drucks vor allem für die Elektrolyse bewährt. Hier konnte aus elektrisch leitfähigem Filament (PLA, Polylactid) eine angepasste Kathode für die Abscheidung elementarer Metalle gedruckt werden. Der elektrische Widerstand hierbei ist mit einigen Ohm zwar deutlich höher als der von vergleichbaren Werkstücken aus Metall, jedoch nicht zu hoch für elektrolytische Abscheidungen.

Durch die Dicke einer solchen ausgedruckten Kathode kann der



Abbildung 2: Runde Kathoden aus PLA.

Widerstand wie auch die Festigkeit dieser bequem angepasst werden. Der gewünschte Durchmesser und damit auch

der Strom pro Fläche kann bequem für jedes Einzelstück angepasst werden.



Abbildung 3: Ausgedruckte grüne Elektrolysezellen mit Vorbild (links).

Für die Verwendung mit radioaktiven Elektrolyten sind ausgedruckte Elektrolysezellen besonders vorteilhaft, da im Falle einer persistenten Kontamination die Zelle kurzerhand ausgetauscht werden kann. Weiterhin konnte so auf einfache Weise Einfluss auf Form und Volumen der Elektrolysezelle genommen werden.

Das häufige Tauschen oder ersetzen 3D gedruckter Werkstücke, weil sie beschädigt, überholt oder aus anderen Gründen obsolet sind, ist aus ökologischer Sicht zwar nicht gänzlich unbedenklich, führt aber nicht zu einer nennenswerten Zunahme der Abfallmenge. Das Polylactid aus dem sie bestehen kann bis auf Zusatzstoffe restlos zu CO₂ verbrannt werden, es gilt außerdem als biologisch abbaubar.^[1]

Die für die Produktion des Filaments eingesetzte Milchsäure entstammt zum größten Teil aus biotechnologischer Herstellung. Die hierfür notwendige Biomasse entstammt dem CO₂-Kreislauf.^[1]

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Erik Strub

| | |
|--|---------|
| Astat in CHCl₃ | 1/2020 |
| Ein schnelles Sandwich mit Technetium | 2/2020 |
| Trägerische Feuerwasser | 4/2020 |
| Information vom Tumor mit DATA | 6/2020 |
| Siamesische Seltenerden | 11/2020 |

| | |
|---|---------|
| Technetium und die sieben Oxidationszustände | 1/2021 |
| Was Astat tat | 2/2021 |
| Ein Mü radioaktiver Stoff | 4/2021 |
| Aller guten Actiniden sind drei | 6/2021 |
| Technetium im Röntgenstrahl | 9/2021 |
| Trithorium | 10/2021 |

Chemie, Köln und Karneval

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5/2020

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E. Strub

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1. ***A quest into molecular-level radiocarbon analysis and an Organic Geochemist's view of the world***
Stephanie Kusch (Universität zu Köln)
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Poster

1. **Separation procedure for ⁴¹Ca AMS samples from heavy concrete**
R. Margreiter, E. Strub, M. Schiffer, A. Stolz, G. Hackenberg, A. Dewald, R. Spanier, M. Dewald
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Abgeschlossene Arbeiten

- 1. Non-invasive analysis of fire gilded layers**
R. Margreiter
Masterthesis
- 2. Weiterentwicklung der Messung von Strontium-90 mittels LSC**
D. Elchine
Bachelorarbeit
- 3. Radiochemische Analyse von bestrahltem Reaktorgraphit**
F. Putkamer
Bachelorarbeit
- 4. Bestimmung von ^7Be und ^{10}Be in Klärschlamm**
J. Laabs
Bachelorarbeit
- 5. Entwicklung einer thermochromatographischen Trennmethode für Mangan aus Chrom**
S. Gülez
Masterarbeit
- 6. Ionenaustauschereigenschaften von MOFs und Koordinationspolymeren**
M. Maslo
Bachelorarbeit
- 7. Charakterisierung des MOFs UoC-3 als Kationenaustauscher für Cäsium**
M. Sadegh Zadeh Farid
Bachelorarbeit
- 8. Korrelation von Pilz- und Bodenkontaminationen von Cs-137**
B. Bir
Projektmodul
- 9. Radionuclides of Beryllium in Sewage Sludge**
J. Kirchhartz
Projekt Modul
- 10. Studies on Rhenium and Technetium compounds with SO_3**
S. Sutorius
Projektmodul
- 11. Development of an undergraduate lab course experiment from an AMS- ^{41}Ca separation procedure**
M. Michel
Projektmodul
- 12. XRF-Messungen an Münzmetallen**
S. Lemke
Bachelorarbeit