28th Seminar Activation Analysis and Gamma Spectroscopy
(SAAGAS 28)

Book of Abstracts

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Provenienz-Analyse von Amphoren aus *Baetica* und *Lusitania*

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In Castro do Vieito, einer frühkaiserzeitlichen römischen Siedlung im Nordwesten Portugals, wurden während archäologischer Rettungsgrabungen in den Jahren 2004 bis 2005 u.a. 14t Keramikscherben sichergestellt. Besonders sog. Haltern-70-Typ-Amphoren wurden in der Antike sehr häufig zur Verschiffung von Gütern wie Wein, Olivenöl oder Garum eingesetzt. Provenienzanalysen an solchen Objeken helfen den Archäologen alte Handelswege zu rekonstruieren.

Ausgewählte Scherben aus Castro do Vieto (CV) sowie von möglichen Produktionsstätten mit Brennöfen in den ehemaligen Provinzen *Baetica* und *Lusitania* wurden mittels XRF, XRD, Mössbauer-Spektroskopie und PGAA analysiert. In dieser Präsentation werden schwerpunktmäßig die PGAA-Ergebnisse sowie die Anwendung multivariaten statistischen Verfahren vorgestellt.

Probenvorbereitung und Bestrahlung

In einer ersten Messkampagne wurden mehr als hundert Scherben aus CV, sowie von acht Brennofenplätzen aus den oben genannten Provinzen mittels PGAA am MLZ analysiert (siehe Karte in *Abb. 1*). Flache rechteckige Stücke von etwa einem halben Zentimeter Dicke und einer Masse von 1-3 g wurden für 1-3 h bestrahlt. Individuelle Messdauern ergaben sich aus einem Schnellcheck der erreichbaren Zählraten.

Mössbauer- und XRD-Messungen wurden jeweils im Physikdepartment in Garching und an der Universität Coimbra durchgeführt. An der NOVA University in Lissabon wurde mittels XRF gemessen.

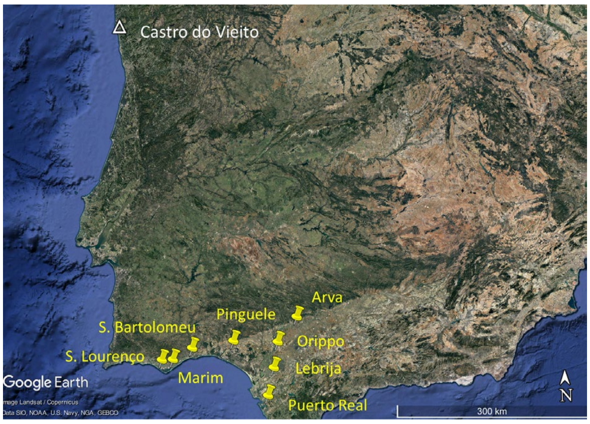

Abbildung 1: Kartenausschnitt der Iberischen Halbinsel mit Lage der Fundstätten. Die Ofenplätze liegen entlang der Flussläufe im Süden.

Ergebnisse und Ausblick

Aus archäologischer Sicht erschien die Annahme, dass die CV-Amphoren aus den zuvor genannten Produktionsstätten entstammen, zunächst schlüssig. Die Ergebnisse der PGAA zeigen jedoch klar, dass dies nicht der Fall ist. In *Abb. 2* ist eine Hauptkomponentenanalyse (PCA) der Proben aus CV und von vier beispielhaften Ofenplätzen dargestellt (zwecks Übersicht - alle anderen separieren ebenso deutlich von CV).

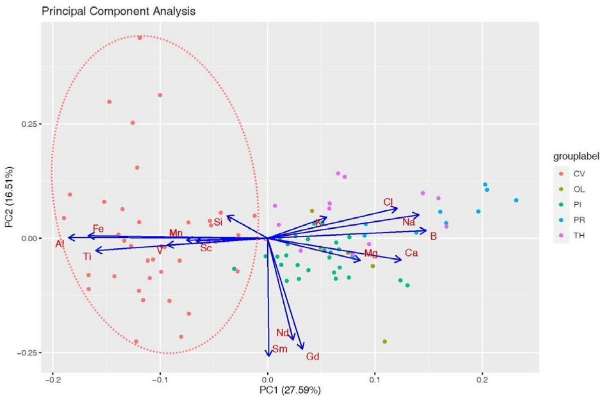

Abbildung 2: PCA von CV und Produktionsstätten (Brennofenplätze).

Zur klaren Unterscheidung zwischen CV und aller übrigen Proben tragen vor allem Hauptbestandteile wie Ca, Fe oder Al bei. Mit einem anderen Set von (Spuren-)Elementen konnten auch Ofenplätze unterschieden werden. Befunde von PGAA, XRF, XRD und Mössbauer stützen sich gegenseitig. Die neue Hypothese lautet nun, dass die Provenienz evtl. nicht in diesem Gebiet liegt und die Amphoren über viel weitere Strecken zum Transport von Handelsguts eingesetzt worden sein könnten. Noch sind nicht alle Stätten analysiert, jedoch reihen sich einige neuere Messungen bestätigend ein. Die bisherigen Befunde sind bereits bzw. werden in Kürze in [1-3] publiziert.

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[1] B. Costa, F. Wagner, W. Häusler, C. Stieghorst, A. Silva, Mössbauer Studies of Haltern 70 Amphorae from Castro do Vieito, Northwest of Portugal. *Crystals* **2024**, *14*(9), 786.

[2] D. Pinheiro, M. Guerra, A. Silva, C. Stieghorst, B. Costa, X-ray compositional microanalysis and X-ray diffraction of Haltern 70 amphorae sherds, *Appl. Phys. A, Spec. Issue: SR2A*, **2025**, in press

[3] C. Stieghorst, B.F. Costa, A. Silva et al., Prompt gamma activation analysis (PGAA) studies of Roman Haltern 70 amphorae from Castro do Vieito and of sherds from the Roman Provinces of Lusitania and Baetica, *in preparation*

Forensik an radioaktiven Proben

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Die Zentrale Technisch-Wissenschaftliche Betriebseinheit Radiochemie München (RCM) der Technischen Universität München befasst sich seit dem sogenannten Transnuklear-Skandal 1987 mit der zerstörungsfreien und der zerstörenden, Charakterisierung von radioaktiven Materialien und Objekten unterschiedlichster Herkunft sowie von Behältern, in denen solche enthalten sind.

Seit 1994 ist die RCM auch im Bereich der nuklearspezifischen Gefahrenabwehr tätig. Als einzige deutsche Institution beteiligt sich die Radiochemie seit 2001 an dem internationalen Ringversuch „CMX“ (Collaborative Materials Exercise) der International-Technical-Working-Group (ITWG). Diese Ringversuche sollen die Zusammenarbeit von Landesbehörden, Strafverfolgung und Analysenlaboren schulen und verbinden die klassische Forensik mit der nuklearspezifischen Gefahrenabwehr.

Durch zahlreiche Projekte, die vor allem durch das Bayerische Staatsministerium für Umwelt und Verbraucherschutz (StMUV) gefördert wurden, konnte bei der RCM die wichtige Zusammenarbeit zwischen den einzelnen Einrichtungen ausgebaut und intensiviert werden.

Ein aktuelles Projekt befasst sich mit der klassischen Forensik an radioaktiven Materialien. Schwerpunkte sind Element-Fingerprints, Röntgen-Durchleuchtung, DNA, Daktyloskopie und Toolmarks. Aufgrund der Radioaktivität der „Asservaten“ ist deren Bearbeitung in normalen Forensik-Laboren nur schwer oder gar nicht möglich.

Ein Ziel der Arbeiten ist die Aufbereitung der Proben in eine Form, die eine Weiterbehandlung in den forensischen Laboren des BLKA ermöglicht unter Berücksichtigung der weiterhin gegebenen gerichtsverwertbaren Verwendung der Proben. Dies beinhaltet auch die Beantwortung der Frage, bis zu welchen Aktivitäten ein Spurensicherungsversuch überhaupt möglich und sinnvoll ist.

Es wird kurz darauf eingegangen, warum die Neutronenaktivierungsanalyse für Proben, wie sie im internationalen Ringversuch „CMX“ betrachtet werden, in der Regel nicht anwendbar ist sowie der aktuelle Stand der Projektarbeiten auf dem Gebiet der Forensik vorgestellt.

Synthesis of a High Specific Activity Radiotracer by Szilárd-Chalmers Methods

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Radiotracers are very useful in industrial detection applications, since the amount of substance needed can be reduced to a minimum, due to the high sensitivity with which ionizing radiation can be detected. By reducing the amount of tracer material introduced, the chemical influence on the system is minimized. The radiotracer chromium(III) tris(2,2,6,6-tetramethyl-3,5-heptanedionate), the chemical structure of which is shown in Figure 1, is already used as a tracer material on a gram scale with an activity of approximately 10 MBq of Cr-51.

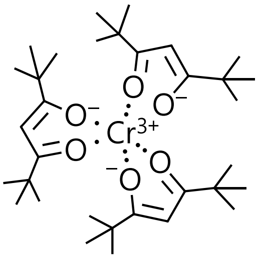

Figure 1: chemical structure of chromium (III) tris(2,2,6,6-tetramethyl-3,5-heptadionate).

To further decrease the amount of substance applied to the test system, research was done to find a method to obtain the same activity but incorporated into only milligram amounts of the complex tracer. In this work, a previously developed downscaled synthesis route for the production of the chromium complex tracer was applied to a highly Cr-51 enriched chromium sample. This sample was produced exploiting the Szilárd-Chalmers effect, by which the molar activity of the substance is increased, through removal of unreacted carrier species after neutron irradiation and thus dramatically decreasing the overall substance amount containing the radiotracer produced. After the isolation of the radioactive residue, the complex synthesis was performed using the chelate effect for introducing the Ligand molecules.[1;2] In Table 1, the chemical and radiochemical results of the final complex synthesis are summarized.

	educt	yield	
substance	5.4×10^{-5} mol	24.7 mg	76.6 %
activity	23.7 MBq	9.2 MBq	71.2 % (1)

Table 1: starting chromium amount for complex synthesis and obtained yield, staring activity of total synthesis and final activity of the product. [1] Decay corrected.

The yield of the desired product was 24.7 mg with an activity of 9.2 MBq, which corresponds to a molar activity of 2.2×10^5 MBq/mol. In Figure 2 the final product of the radiotracer is pictured, which was characterized by TLC analysis relative to an authentic sample.

Figure 2: synthesized and purified radiotracer.

[1] H. F. Holtzclaw, J. P. Fackler, B. E. Douglas, J. H. Worrell, S. L. Holt; S. Kirschner, *Inorganic Synthesis* **1986,** V.24, 183-184.

[2] M.A.K. Ahmed, H. Fjellvåg, A. Prof. Dr. Kjekshus, R.K. Birkedal, P. Norby, D.S. Wragg, N.S. Gupta, Syntheses, Crystal Structures, and Thermal Stabilities of Polymorphs of Cr(thd)3 *ZAAC* **2010**, 636(13-14), 2422-2432 DOI: 10.1002/zaac.201000160

Progress in Ru-103 Radiolabelling of Elastomers

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Mechanical degradation of rubber significantly affects its performance and lifetime, making it a critical factor in the field of industrial applications [1]. Ethylene-propylene-diene terpolymer (EPDM) rubber is widely used in automotive parts and industrial seals due to its chemical resistance and flexibility. However, it is susceptible to abrasive degradation, so tracking this process, especially in real time, is crucial. Radiolabelling EPDM with gamma emitting radioactive isotopes offers a powerful analytical method to trace and monitor this degradation, as even minimal radiotracer amounts may still enable easy and precise detection [2,3].

In this study, Ru-103 was selected based on its suitable half-life (~ 40 d) and gamma emission (497 keV, 91 %) properties. The radiolabelling process was conducted by diffusing gaseous Ru-103 labelled ruthenium(VIII) oxide into the EPDM matrix. Subsequent reaction with unsaturations within the rubber immobilizes the ruthenium-based tracer. Gamma spectrometry and autoradiographic methods were employed to confirm the successful incorporation, assess spatial distribution of the radiotracer within the EPDM matrix and confirm irreversibility of the labelling process.

Gamma spectroscopic and autoradiographic results affirm that EPDM can be very effectively radiolabelled, and the amount of radioactivity within the rubber can be evaluated and controlled by several different parameters. Labelling depth is controlled principally by exposure time (to RuO_4) and the distribution of Ru-103 in the labelled EPDM is very nearly homogeneous (no measurable concentration gradient). To ensure that the radioactivity is not confined to surface deposition, solvent (acetone) treatments using ultrasonic cleaning procedures were performed, and the spatial distribution of the activity inside the rubber was visualized showing minimal loss of activity on treatment.

Further research will focus on characterizing and quantifying the diffusion of Ru-103 into the crosslinked rubber matrix at the molecular level.

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[1] D. Stamenkovic et al, *Tribol Ind* **2014**, 36, 9-16

[2] J. Sterba, *J. Radioanal. Nucl. Chem.* **2018**, 316, 753-759

[3] G. Steinhauser et al, *Environ Sci Pollut Res Int* **2013**, 20, 2527-2534

Gammaspektrometrie an einem mit Cs-137 kontaminierten Schottersimulat

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Die Repräsentativität der Beprobung von radioaktiven Abfallstoffen wird stark durch Inhomogenitäten im Material beeinflusst. Da Abfall physikalisch sowie chemisch äußerst divers sein kann und verschiedene Radioisotope enthält, ist eine repräsentative Probennahme schwierig. In dieser Arbeit werden Methoden untersucht, um inhomogene Aktivitätskonzentrationen zu quantifizieren. Weiters werden Versuche unternommen, diese zu minimieren und mögliche Implementierungen zum Routinebetrieb darzulegen.

Methoden

Für die Aktivitätsmessungen wird ein HPGe-Detektor von Canberra verwendet. Im Allgemeinen wird für die Arbeit ein gewöhnlicher Schotter, hauptsächlich aus Quarz und Kalk bestehend, vom Gelände der NES herangezogen. Er enthält Partikel bis zu einer Größe von 5 mm. Der Schotter wurde zuvor mit einer Cs-137-Lösung versetzt und luftgetrocknet und enthält eine bekannte Aktivität durch Cs-137.

Stichprobenartige Probennahmen und gammaspektrometrische Analysen zeigen trotz der einheitlichen Kontamination signifikante Abweichungen der Aktivitätskonzentration untereinander.

Diese Inhomogenitäten werden mittels Gammaspektrometrie quantifiziert. Dabei wird zum einen mit gezielter Probennahme untersucht, ob und in welchem Ausmaß ein Tiefenprofil während der Kontamination entstanden ist. Zum anderen werden die Partikel mittels Siebanalysen in Fraktionen mit festen Größenintervallen separiert und anschließend gammaspektrometrisch gemessen.

Ergänzend zur Gammaspektrometrie werden dieselben Proben durch Röntgenfluoreszenzanalyse auf ihre elementare Zusammensetzung untersucht. Gesucht werden Abweichungen in den Elementarkonzentrationen, da Mineralien unterschiedliches Adsorptionsverhalten von Cäsium aufweisen [2-4].

Ergebnisse der Siebanalyse

Man weiß aufgrund bisheriger Untersuchungen mit Erde [1], dass die absolute Oberfläche eines Analyten Auswirkungen auf dessen Aktivitätskonzentration hat. Die spezifische Oberfläche der Fraktionen wurde berechnet und basiert auf der Annahme, dass der Schotter aus kugelförmigen Partikeln besteht und jeder Fraktion eine mittlere Partikelgröße zugeordnet werden kann. Die Siebanalysen zeigen, dass die Aktivität direkt im Zusammenhang steht zur spezifischen Oberfläche der Fraktionen (siehe Abb. 1). Dieses Ergebnis wird von zwei Probensätzen, die gleich und unabhängig voneinander behandelt wurden, verifiziert.

Mit weiteren Messmethoden wie zum Beispiel der BET-Methode könnte die Oberfläche direkt gemessen und genau bestimmt werden.

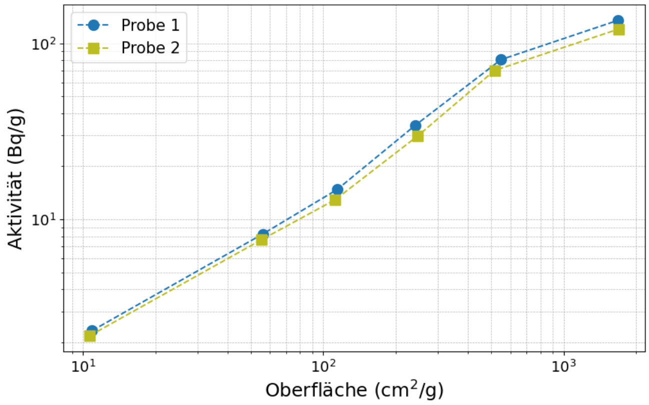

Abbildung 1: Gemessene Aktivitätskonzentrationen aus der Gammaspektrometrie im Vergleich mit der spezifischen Oberfläche. Die Oberfläche wurde mit einem angenommenen mittleren Durchmesser innerhalb der Fraktionen berechnet.

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[1] G. Wurmbauer, Diplomarbeit, Universität Wien, **2004**, 55-65.

[2] R. M. Cornell, *Adsorption of cesium on minerals: A review*. Journal of Radioanalytical and Nuclear Chemistry, Articles vol. 171, **1993**.

[3] C. Hsu, *Sorption and Desorption Behavior of Cesium on Soil Components*, Vol. 45, Appl. Rodiaf. Isor., **1994**.

[4] J. H. Sterba, H. Sperrer, F. Wallenko, J.M. Welch, *Adsorption characteristics of a clinoptilolite-rich zeolite compound for Sr and Cs*, Journal of Radioanalytical and Nuclear Chemistry 318, 267–270, **2018**.

Natürlich oder künstlich? Ein Beitrag eines ISO-zertifizierten Gamma-Messplatzes zum Verbrauchschutz in der Edelsteinbranche

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Heutzutage werden fast alle verkauften Edelsteine in irgendeiner Form behandelt [1]. Neben konventionellen Methoden ist die Bestrahlung eine der effektivsten Methoden, um bei bestimmten Edelsteintypen die Farbintensität und die optischen Eigenschaften zu verbessern. Jährlich werden so tonnenweise Edelsteine bestrahlt. Um die gesetzlichen Deklarationspflichten einzuhalten, ist ein Nachweis dieser Behandlungen von essenzieller Bedeutung.

Aufgrund des höheren Aufwands und der längeren Abklingzeit bei der Neutronenbestrahlung werden zunehmend Edelsteine mit hochenergetischen Elektronen in Beschleunigern bestrahlt. Während neutronenbestrahlte Edelsteine aufgrund zahlreicher, insbesondere langlebiger, radioaktiver Aktivierungsprodukte von Spurenelementen leicht durch γ-Spektroskopie identifiziert werden können, stellt die Identifizierung einer Elektronenbestrahlung eine Herausforderung dar, da hier kaum Aktivierungen zu erwarten sind.

In Zusammenarbeit mit der Deutschen Gemmologischen Gesellschaft e.V. (DGemG) wurde am Messlabor des Forschungsreaktors FRM II eine Studie an bestrahlten Morganiten – einer Beryll-Varietät – durchgeführt [2]. Die Steinproben aus verschiedenen Herkunftsländern wurden zunächst halbiert. Jeweils eine Hälfte mit einem Gewicht von weniger als 5 Karat (1 g) wurde an einem kommerziellen Beschleuniger unter Standardbedingungen mit etwa 10 MeV Elektronen bis zu 250 MGy bestrahlt. Die durch die Bestrahlung hervorgerufene Farbänderung ist in Abbildung 1 deutlich erkennbar.

Anschließend wurden die Proben, sowohl einzeln als auch in Gruppen, am FRM II etwa 24 Stunden lang mit einem HPGe-Detektor mit einer relativen Effizienz von 40 % gemessen. Der Gammamessplatz ist seit 2022 nach ISO 9001 zertifiziert [3] und wird in der Präsentation kurz vorgestellt.

Abbildung 1: 15 Morganitenproben, jeweils links: unbestrahlt, rechts: mit 10 MeV Elektronen bestrahlt.

Die ersten Messungen erfolgten nach einer Abklingzeit von etwa drei Wochen. Nach einer thermischen Behandlung der Proben bei bis zu 400 °C im Labor der DGemG wurden nach 100 Tagen zweite Messungen durchgeführt. Zur Kontrolle der natürlichen Aktivität in den Proben wurden die unbehandelten Hälften aller Proben ebenfalls gamma-spektroskopisch untersucht.

In allen bestrahlten Proben wurden die radioaktiven Isotope ^{132}Cs und ^{134}Cs nachgewiesen, wobei das kurzlebigere ^{132}Cs ($T_{1/2}$ = 6,47 d) bei den zweiten Messungen bereits vollständig abgeklungen war. Die Aktivitäten waren zwar sehr schwach, lagen jedoch deutlich über den Nachweisgrenzen. Die Ergebnisse in Tabelle 1 zeigen eine eindeutige Korrelation zwischen dem Cs-Gehalt und der spezifischen Aktivität beider Cs-Isotope.

Die Bildungsprozesse der radioaktiven Nuklide wurden diskutiert. Neben einer primären Photonenaktivierung könnte eine sekundäre Neutronenaktivierung eine Rolle gespielt haben. Beryllium ist ein Hauptelement in der Beryllstruktur, weshalb durch die (γ,n)-Reaktion eine Neutronenproduktion durchaus relevant war.

Das langlebige Isotop ^{134}Cs könnte als Indikator für die Elektronenbestrahlung von Morganiten dienen. Mit seiner Halbwertszeit von etwa zwei Jahren können auch viele Jahre nach der Behandlung bestrahlte Edelsteine identifiziert werden.

Auch in den folgenden (laufenden) Studien konnten weitere künstliche radioaktive Isotope wie ^{152}Eu und ^{22}Na in Edelsteinen detektiert werden, die mit ionisierender Strahlung behandelt wurden.

Probe	Gewicht	Cs_2O* (*wt.* %)	spez. Aktivität (Bq/g)	
			^{132}Cs	^{134}Cs
Braz1	0.78 g	8.76 ± 0.14	74 ± 4	0.31 ± 0.02
Mada1	0.36 g	8.59 ± 0.15	57 ± 3	0.26 ± 0.04
Moz1	0.85 g	0.72 ± 0.04	5.5 ± 0.4	0.045 ± 0.008
Moz3	0.40 g	0.74 ± 0.04	5.5 ± 0.4	0.045 ± 0.008
Nig8	0.39 g	5.43 ± 0.11	24 ± 2	0.15 ± 0.02

Tabelle 1: spezifische Aktivität von Cs-Isotopen in den bestrahlten Morganitenproben. *: Die chemischen Zusammensetzungen in einzelnen Proben wurden von der DGemG veranlasst.

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[1] https://thenaturalgem.com/, (Jan. **2025**)

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[3] X. Li, V. Hutanu, H. Schulz, F. Jeschke, A. Pichlmaier, in Proc. RRFM, Antwerp, Belgium, **2023**, pp. 501-507.

Validation of gamma spectrometry method for the characterization of irradiated reactor graphite from German research reactors

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Proceedings

Graphite is a frequently used material in various types of nuclear reactors, as construction material, moderators, reflectors and fuel sleeving material. Over time the graphite and its impurities undergo activation by neutron capture processes. Various radionuclides are being produced including gamma emitters such as Co-60 and Eu-152 as well as pure beta emitters such as H-3 and C-14. Due to the long half-life of C-14 and the possible releasing behaviour of H-3 and C-14 under disposal conditions, direct disposal without any pre-treatment is not an option [1].

In the process of dismantling, the gamma-ray emitting radionuclides may be responsible for significant dose rates, leading to the necessity of special handling. Therefore, a comprehensive characterization of the graphite´s radionuclide inventory is of crucial importance. Treatment steps of irradiated graphite may be packaging it in suitable containers. An alternative can be decontamination to reduce the dose rate or to recycle the graphite for further use in nuclear facilities. The project i-GraDe (funded by BMBF, 15S9442) aims to develop suitable characterization and decontamination methods for irradiated nuclear graphite.

Gamma-ray spectrometry with HPGe detectors is commonly used to identify and quantify gamma emitters. This technique has several advantages: being non-destructive due to the strong penetration of the gamma ray through matter, little sample preparation is needed, and any sample geometry can be measured. In order to obtain representative activity values, efficiency curves for all used sample geometries are essential. Commonly, multi-nuclide standards covering a wide energy range are used. Moreover, MC-based simulation software such as EFFTRAN [2], can be used to adapt the measured efficiency curves to other suitable geometries of samples. In principle, these software codes allow the prediction of the efficiency curves of any geometry. Nevertheless, the simulation software has primarily to be validated using standard samples with a known activity. As shown on Figure 1 the experimental efficiency curve of a sample is compared to the simulated efficiency curve produced from a standard sample.

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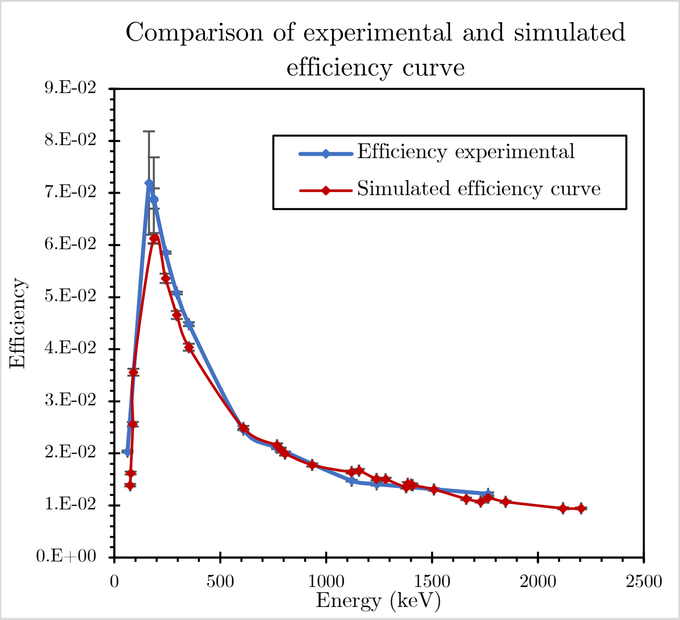

Figure 1: Efficiency curves obtain with experimental and simulation data

However, it is not possible to measure pure beta emitters with HPGe detectors, so measurements must be carried out using a liquid scintillation counter. As this method requires a destructive sample preparation method and only a small amount of the samples can be analysed, the homogeneity of the radionuclides within the sample must be determined for representative sampling. This is carried out using a modern digital autoradiography device, which is calibrated beforehand to the individual nuclides in a specific activity range. This provides crucial insight in the spatial distribution of the radionuclides but also enables qualitative and quantitative analyses of the beta emitters in the graphite.
This presentation will focus on the validation of the MC-based simulation software for determining the efficiency curves of the HPGe detectors.

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Evaluating the Ion-exchange Capacity of a Specially Prepared Zeolite

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Zeolites, ion-exchangers [1] that can be mined in nature, have applications in agriculture, waste disposal, water purification and environmental decontamination. Their ion-exchange capacity and selectivity depend on the cation-accessible sites within their crystalline pore structures. The ion-exchange properties make them particularly relevant for mitigating radioactive contamination, such as caesium (Cs^+) and strontium (Sr^{2+}), following nuclear reactor incidents [2].

This study aims to evaluate the exchange capacity of a specially prepared zeolite (purified clinoptilolite-tuff, PCT) for Cs^+ and Sr^{2+} and other physiologically relevant ions. Traditional radioanalytical methods based on radiotracers and gamma spectroscopy have provided valuable insights into the exchange dynamics of PCT [2]. The ion-exchange capacity has been tested by applying solutions with different ion concentrations to the PCT. The samples gathered after the PCT-solution ion-exchange and reference samples taken before the contact with the PCT are measured using a gamma counter with the samples in the same geometry. The activity measured is used to calculate the ion concentration in each sample. The concentration difference between the reference sample and the ion-exchanged sample gives the amount exchanged. Finally, the ion-exchange capacity can be evaluated by determining the number of ions exchanged relative to their applied concentrations.

Furthermore, this research introduces ion chromatography [3] as a complement to radioanalytical methods. Ion chromatography uses different ions' conductivity to determine the PCT's ion-exchange capacity. A key advantage of combining ion chromatography with gamma spectroscopy is its ability to simultaneously quantify inactive ions such as calcium released from the zeolite, corresponding to the cation-accessible sites, and the number of ions exchanged into the PCT.

Understanding the PCT ion-exchange capacity for different ions will have an impact on evaluating the potential of the PCT in various applications like environmental or wound decontamination.

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Elektroneneinfang von ^{98}Tc

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Für ^{100}Tc wurde sowohl der Elektroneneinfang (EC) als auch der β^--Zerfall nachgewiesen. Die Vermutung, dass auch das neutronenärmere ^{98}Tc einen EC-Zweig besitzen sollte, ist also naheliegend. Auch eine nähere Betrachtung der Niveauschemata der Zerfallstöchter ^{98}Ru bzw. ^{98}Mo legt das nahe: In beiden Fällen ist die Zerfallsenergie ausreichend, um aus dem 6+-Zustand des ^{98}Tc einen 4+-Zustand zu bevölkern, der sich jeweils über die Emission von 2 Photonen über einen 2+-Zustand in den 0+-Grundzustand abregen kann [1]. Ein Versuch von Kobayashi [2], die entsprechenden Übergänge von 722 keV und 787 keV nachzuweisen, lieferte allerdings nur eine Abschätzung der oberen Grenze von < 1 % für den EC, was später durch unsere eigenen Messungen reproduziert werden konnte [3].

Messung des Zerfalls von ^{98}Tc

^{98}Tc kann nur schwer als isotopenrein in Kernreaktionen erhalten werden. Insbesondere ist es bei der Kernspaltung durch das stabile ^{98}Mo, seinem vermuteten Zerfallsprodukt, von der Isobarenkette mit A=98 abgeschirmt. ^{99}Tc-Proben sind jedoch in ausreichender Menge von mehreren Gramm verfügbar. Diese enthalten stets auch einen kleinen Anteil (< 1 ppm) ^{99}Tc als primäres Spaltprodukt.

Eine Probe von ca. 3 g $KTcO_4$ (ca. 2 GBq) wurde für mehrere Wochen am Clover-Detektor-Messplatz des Instituts für Kernphysik gemessen [4]. Die Probe befand sich dafür in einem Bleigefäß, um die β-Strahlung die 89.5-keV Linie des ^{99}Tc und Röntgen-Strahlung weitgehend abzuschirmen. Dadurch konnte die Totzeit des Systems reduziert werden

In den resultierenden γ-Spektren (s. Abbildung 1) findet sich sowohl die Signatur des ^{98}Tc β—Zerfalls, nämlich die γ-Linien bei 745 keV und 653 keV, die aus den 4+⟶2+ und 2+⟶0+ Übergängen in ^{98}Ru hervorgehen

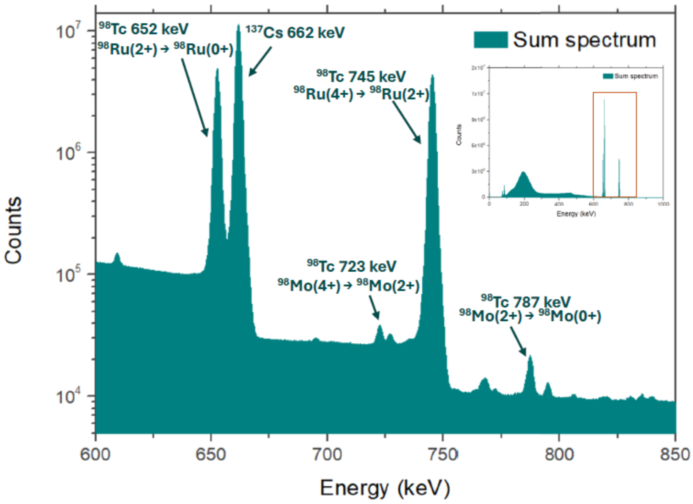

Abbildung 1: Ausschnitt des gemessenen γ-Spektrums. Markiert sind die 4+⟶2+ und 2+⟶0+ Übergänge in den Tochterisotopen des β—Zerfalls (^{98}Ru) und des EC (^{98}Mo)

Analog konnten nun erstmals auch die 4+⟶2+ und 2+⟶0+ Übergängen in ^{98}Mo gemessen und quantifiziert werden. Wie erwartet, sind die relativen Intensitäten 1:1, und aus den Messergebnissen lässt sich ein Verzweigungsverhältnis von ca. 0.27 % +/- 0.04 % für den Elektroneneinfangzweig berechnen.

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Cosmogenic radionuclides in meteorites – Reloaded – by Instrumental Accelerator Mass Spectrometry (IAMS)

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Introduction

Long-lived radionuclides in meteorites are a result of the interaction with cosmic rays. These cosmogenic nuclides (CNs) record the history of extraterrestrial matter (Fig. 1). To reconstruct this history, the parameters of interest are:

1. pre-atmospheric size of the small body in space (meteoroid) and shielding depth of sample;
2. irradiation time in space;
3. potential complex exposure, i.e., repeated collisions or inherited CNs from pre-exposure at the surface of the meteoroid's parent body (asteroid, Moon, Mars);
4. residence time on Earth, i.e., terrestrial age.

Accelerator mass spectrometry (AMS) is the preferred method for the detection of CNs such as ^{10}Be, ^{14}C, ^{26}Al, ^{36}Cl, and ^{41}Ca ($t_{1/2}$=6 ka-1.4 Ma). However, until recently tedious radiochemical separation to deplete matrices and isobars has been essential for AMS preventing rapid analysis.

Now, the unique *Ion-Laser InterAction Mass Spectrometry* (ILIAMS) system at the Vienna Environmental Research Accelerator (VERA) provides isobar suppression of up to 14 orders of magnitude [1,2]. Thus, ILIAMS-assisted AMS enables the direct detection of ^{26}Al/^{27}Al (~10^{-10}) and ^{41}Ca/^{40}Ca (~10^{-12}) in stony meteorite samples as small as 1 mg. Stable ion currents originate from the intrinsic Al and Ca (each ~1%), while isobars of the naturally-abundant elements (15-20% Mg, ~1‰ K) do not interfere anymore with CN detection. Hence, the method, making radiochemical separation redundant, has been named *Instrumental AMS* (IAMS).

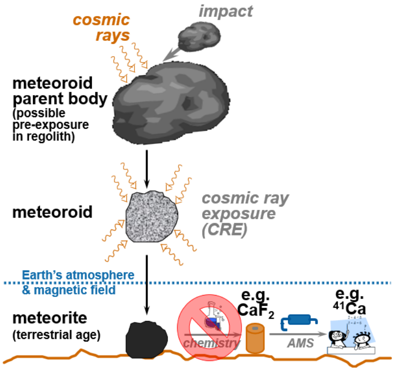

Figure 1: Schematic of the history of a meteorite in space, on Earth, and in the VERA laboratory for AMS of cosmogenic radionuclides.

For iron meteorites, chemistry is still needed due to the absence of native Al and Ca. However, ILIAMS facilitates straightforward isobar suppression and remarkably efficient ^{36}Cl, ^{41}Ca, and ^{26}Al determination; the latter by high current of AlO^- (instead of Al^-). Other CNs (^{44}Ti, ^{53}Mn, ^{59}Ni; $t_{1/2}$=60 a-3.7 Ma) are currently under AMS development at VERA.

Exemplary results

We have investigated over 25 meteorite samples for ^{26}Al and ^{41}Ca by IAMS; with the most notable cases being the Austrian, French, and German meteorite falls from 2023/2024 Elmshorn [3], Haag, Ribbeck [4], and St.-Pierre-le-Viger. In addition, the destructive IAMS method (sample size: ~1 mg) was validated through comparison with non-destructive γ-spectrometry in the underground laboratory Felsenkeller [5] of the VKTA Rossendorf (sample size: 20 g-3.7 kg) (Fig. 2).

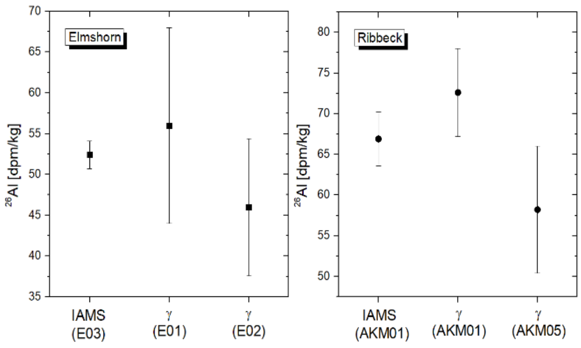

Figure 2: IAMS and γ-spectrometry ^{26}Al data for different meteorite pieces of Elmshorn [3] and Ribbeck [4], respectively.

Furthermore, eleven samples have been identified as meteor-wrongs, i.e., those samples do not contain CNs at a level expected for materials being exposed to cosmic radiation for typical exposure times of stony meteorites.

Outlook

At present, the bulk ^{27}Al and ^{40}Ca data from collaborators' analyses is being used to convert nuclide ratios into specific radionuclide activities (disintegrations min^{-1} kg^{-1}). However, a new setup is being installed at VERA to measure these elements ourselves in the identical powder by non-destructive Particle-Induced X-ray Emission (PIXE) in the near future.

Acknowledgments

We thank A. Bischoff, D. Heinlein, L. Ferrière, A. Patzek, M. Patzek, L. Pittarello et al. for precious samples, the VERA cosmo team (P. Steier, C. Vivo Vilches) and master students D. Krebs and D. Ibrahimovic, for assisting in AMS, and ChETEC-INFRA (EU Horizon 2020; No. 101008324) for €.

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The INAA facility in Delft: Fully automated, high capacity, high accuracy and opportunities for transnational access

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The INAA facility at the Reactor Institute of the Delft University of Technology is built on over 50 years of experience in automation of gamma-ray software for spectrum analysis and interpretation, automation of gamma-ray spectrometers by sample changers and integrating this all with quality assurance and quality (validity) control. The facility comprises 2 spectrometers with coaxial detectors and 3 spectrometers with well-type detectors, and a spectrometer with the fast rabbit system. The system allows for the simultaneous use of the various detectors by different projects which speeds up the turnaround time for each project; rather than having the measurements running sequential on one or two spectrometers.

The typical capacity is about 14 samples, 20 flux monitors and 2 quality control samples per spectrometer per day, resulting in ca. 100 samples/day if only 1 measurement is needed, and ca. 50 samples /day if 2 measurements (e.g., after 1 and 3 weeks decay time) are desired . The fast rabbit system is operated manually, and the number of samples to be analysed depends on the half-life dependent protocol but is typically also in the order of several tens per day.

Taking advantage of a research reactor with a reliable operating schedule of about 40 weeks/year at about 100 hours/week, the system provides a capacity of several thousands of samples for multi-element analysis a year.

The gamma-ray software can be run in the fully automated mode without operator intervention or, in and interactive mode, The software is largely similar to the IAEA -k0 software but spectra can also be made available in other formats to be analysed by software such as GammaVision, Genie2000. These options facilitate irradiations and measurements at the facility and (remote) analysis by user-specific software.

The capacity of the INAA facility in Delft can thus be made available for projects from users worldwide, e.g. for other NAA facilities dealing with capacity limitations by lack of automation, limited reactor availability or technical difficulties. Sample preparation and spectrum analysis can done at the end-user's facility whereas the irradiations and measurements at contracted out to the Delft Facility at a nominal fee.

Manganese metabolism in photosynthetic tissues

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Photosynthesis has a prime importance on Earth, as it contributes significantly to the atmosphere, climate, and the evolution of life, including humans. While photosystem (PS) I is the major sink for iron, the function of PSII responsible for the photocatalyzed oxygen evolution relies on the manganese, as Mn_4CaO_5 cluster is involved in the water splitting complex [1]. PSII forms super- and megacomplexes in the thylakoids in order to optimise the effective utilisation of light. Though Mn is essential for PSII formation, little is known about the light dependence of plastidial Mn uptake and how Mn deficiency is managed in photosynthetic cells. In the present work Mn uptake and incorporation was investigated on two model plant systems: sugar beet (*Beta vulgaris* L. cv. Orbis) and liverwort (*Marchantia polymorpha* L. Tak-1 line, Fig. 1A).

Neutron activation analysis (NAA) of the chloroplasts suspensions accompanied by X-band electron paramagnetic resonance (EPR) spectroscopy provided information not only about the concentration of Mn but also about the Mn speciation. Comparison of samples obtained from the chloroplasts stored in different light conditions and supplied by different Mn solutions suggested that Mn uptake is light triggered and is immediately incorporated into the Mn_4CaO_5 cluster. Higher concentration of Mn supply (>500 µM) saturates the system and leads to accumulation of Mn^{2+} between the two envelope membranes.

Particle-induced X-ray emission (PIXE) analysis of cryosectioned, lyophilised thalli showed that in the case Mn sufficient supply the Kα signal of Mn is detected in similar intensities in all cell types, while Fe Kα is located in photosynthetically active upper and lower cell layers. Under Mn deficiency, the mapping showed higher Mn-Kα signal in in the lower cell layer, suggesting that the allocation of Mn is directed towards the photosynthetically active cells. Mn deficiency leads to the residual Mn content of the thalli to be primarily allocated towards the youngest thallus parts of developing photosynthetic activity, coupled with premature senescence in the older thallus segments.

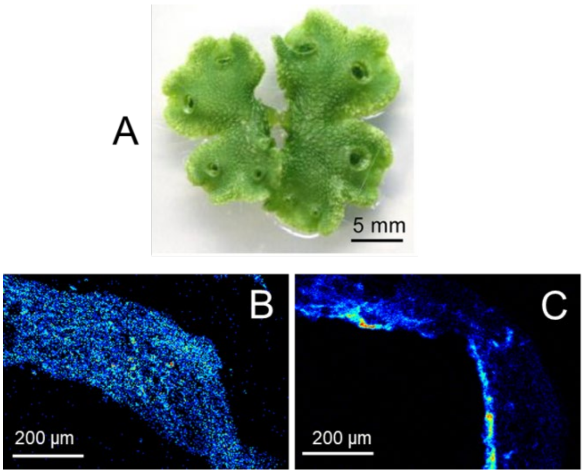

Figure 1: The picture of tissue of *Marchantia polymorpha* (A) [2] and particle-induced X-ray emission images on the cross-section Mn-K*α* signal intensity distribution in Mn sufficient (B) and Mn deficient (C) thalli.

This work was supported by the grant K-146865 of National Research, Development and Innovation Office, Hungary (NKFIH), by the Bilateral Research Agreement of Centre National de la Recherche Scientifique and Hungarian Academy of Sciences. The works was funded under ReMade@ARI PID 27548 by the European Union as part of the Horizon Europe call HORIZON-INFRA-2021-SERV-01 under grant agreement number 101058414 and co-funded by UK Research and Innovation under the UK government's Horizon Europe funding guarantee (grant number 10039728) and by the Swiss State Secretariat for Education, Research and Innovation under contract number 22.0018.

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Can we use 3D printed sample holders in Neutron Activation Analysis?

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The potential of additive manufacturing to produce low-cost devices in a faster, simplified process than traditional methods is increasingly being exploited in many areas of science and also in nuclear industry [1-3]. The NAA community is searching for a radiation-resistant, easily manufacturable sample holder, for which 3D printing has the potential to provide a solution. The primary priority is to identify raw materials that exhibit resistance to radiation, with the additional advantages of ease of decontamination and, therefore, presumed acid and heat resistance. A wide variety of 3D printing plastics are available, each exhibiting well-defined physical properties. However, the chemical behaviour of these plastics, particularly in terms of radiation resistance and activation property characterisation, falls significantly short of expectations.

PEEK (polyether-ether-ketone), PEKK (polyether-ketone-ketone), PEI (polyether-imide), and other high-performance thermoplastics had been proposed for testing by the material expert of a 3D printing company, as these materials have high heat resistance, and low coefficient of thermal expansion, they also have good chemical resistance and flame retardance. Variety of raw materials (see Fig. 1) were irradiated for shorter and longer term at the Budapest Research Reactor and the activity of the samples were measured in the BNC-NAA laboratory (see Fig. 2). Activation properties were measured, colour changes were visualized, and the purity of the material was investigated serving information on trace element content and residual activity. Even traces of some elements in the material makes a problem to handle the sample immediately after irradiation, some can cause increased residual activity.

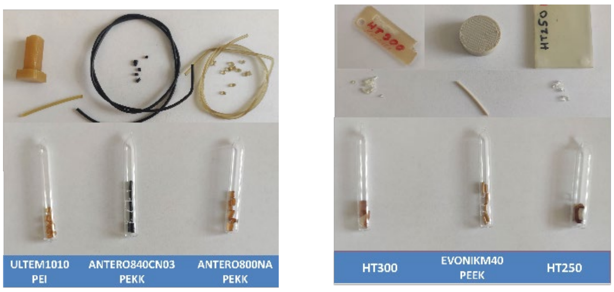

Figure 1: Different type or resins were irradiated for shorter and longer time periods.

A PEI resin, Stratasys ULTEM 1010, appeared to be the purest material and therefore the most suitable for use as a sample holder for NAA. All detectable elements in it are below mg/kg level, however it changes colour, and gets a bit darker after long irradiation. There is another Stratasys ULTEM PEI number 9085 but it has high Cl content.

A PEKK type resin Stratasys Antero 840 CN 03 has also relatively high Cl, but also Fe, and Co content, while Stratasys Antero 800 NA is more suitable candidate but rather for long irradiation (see Fig. 2).

Two high temperature resistant raw materials (HT250 and HT300) showed also colour changes after irradiation, but HT 250 would be a reasonable sample holder for short (max 10 minutes) irradiations (see Fig. 2).

A PEEK is considered one of the best-performing, semi-crystalline thermoplastics available today in industry, but for example EvonikM40, which does not show visible colour changes due to irradiation, it gets highly active, as it has high Na and Mg content, so it found to be inadequate as a sample holder. A PEEK is widely regarded as one of the most effective semi-crystalline thermoplastics currently employed in industry. EvonikM40, for instance, exhibits minimal colour change upon irradiation, however, due to its high sodium (Na) and magnesium (Mg) content, leading to its notable activity (see Fig. 2), its use as a sample holder is deemed unsatisfactory.

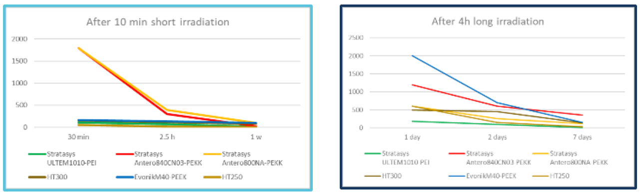

Figure 2: Activity decrease with time after short (10 minutes) and longer (4 hours) irradiation.

Composition and activity of those 3D printable raw materials which seemed to be suitable for some kind of irradiation were measured with NAA before and after 3D printing. The printing of these materials requires high-temperature processing, which effects the volatile element content of the examined sample.

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Der Forschungsreaktor TRIGA Mainz

Klaus Eberhardt, Christopher Geppert

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Der Forschungsreaktor TRIGA Mainz [1] ist einer von nur zwei Forschungsreaktoren in Deutschland. Mit dem Einsetzen des 57. Brennelements in den Reaktorkern wurde der Reaktor am 3. August 1965 erstmals kritisch. Bei der Einweihungsfeier im April 1967 erfolgte die offizielle Inbetriebnahme durch den Nobelpreisträger und Entdecker der Kernspaltung Otto Hahn. Seitdem ist der Reaktor bis auf eine kurze Umbauphase im Jahr 1995 störungsfrei in Betrieb. Die Nutzung des TRIGA Mainz beruht auf drei Säulen: (i) Grundlagenforschung in Kernchemie und -physik; (ii) angewandte Forschung in den Bereichen Chemie, Physik, Material- und Lebenswissenschaften; (iii) die Ausbildung von Studierenden und Wissenschaftlern und der Kompetenzerhalt in den Bereichen Radioaktivität und Strahlenschutz durch eine Vielzahl von Kursen für Teilnehmer aus den Bereichen Naturwissenschaft, Technik und Medizin. In allen diesen Bereichen spielt der TRIGA-Reaktor als intensive Neutronenquelle und Trainingsreaktor eine unverzichtbare Rolle auf nationaler und internationaler Ebene.

Aufgrund der speziellen Zusammensetzung des Brennstoffs weisen Reaktoren vom TRIGA-Typ [2] durch ihren großen prompten negativen Temperaturkoeffizienten ein sehr hohes Maß an inhärenter Sicherheit auf. Es ist daher möglich, sie zu pulsen, d. h. kurzfristig auf eine sehr hohe Leistung zu bringen. Der TRIGA Mainz kann im Dauerbetrieb sehr flexibel mit Leistungen zwischen 100 mW und 100 kW gefahren werden. Ebenfalls möglich ist ein Pulsbetrieb, bei dem kurzzeitig (für ca. 30 ms) die Leistung auf bis zu 200 MW ansteigt. Mit steigender Brennstofftemperatur wird die Leistungsexkursion durch den prompten negativen Temperaturkoeffizienten gebremst und auf einen Gleichgewichtszustand zurückgeführt. Die Neutronenfluenz dieser Impulse kann zur Erzeugung kurzlebiger Nuklide für die Aktivierungsanalyse oder zur Herstellung kurzlebiger Spaltprodukte mit Vorteil ausgenutzt werden. Vergleicht man für Nuklide mit verschiedenen Halbwertszeiten (HWZ) die Sättigungsaktivität bei einer Dauerbestrahlung mit der Aktivität bei einer Pulsbestrahlung ergibt sich für HWZ kleiner 55 s ein Vorteil bei Pulsbestrahlungen. Dies eröffnet spezielle Möglichkeiten für die Neutronenaktivierungsanalyse mit kurzlebigen Nukliden [3,4].

Abbildung 1 zeigt einen vertikalen und einen horizontalen Querschnitt des Reaktors. Der TRIGA Mainz ist mit der gesamten Abschirm-Konstruktion über der Erde errichtet. Vier horizontale Strahlrohre und eine thermische Säule durchdringen die Betonabschirmung und reichen bis an den Reflektor bzw. Reaktorkern. Die thermische Säule wird durch ein fahrbares Beton-Strahlenschutztor abgeschlossen.
Ein rotierendes Bestrahlungskarussell im Oberteil des Graphitreflektors erlaubt die Bestrahlung von bis zu 80 Proben in 40 Positionen. Im zentralen Bestrahlungsrohr können Proben bei maximalem thermischen und schnellem Neutronenfluss bestrahlt werden. Weiterhin existieren zwei Rohrpostanlagen, mit denen Proben in wenigen Sekunden in den Reaktorkern eingebracht bzw. aus ihm entnommen werden können, ohne den Reaktorbetrieb zu unterbrechen.

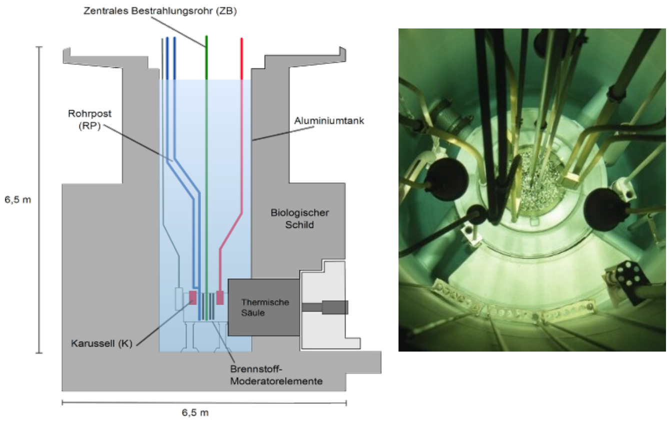

Abbildung 1: Aufbau des TRIGA Mainz mit Bestrahlungseinrichtungen (links) und Blick auf den Reaktorkern (rechts).

Der Beitrag beschreibt den Aufbau und die Anwendungen des TRIGA Mainz, insbesondere im Hinblick auf Bestrahlungsmöglichkeiten für die Neutronenaktivierungsanalyse.

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Neutronenaktivierungsanalysen am Forschungsreaktor TRIGA Mainz

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Der Forschungsreaktor TRIGA Mainz [1, 2], eine zentrale Einrichtung der Johannes Gutenberg-Universität (JGU), bietet die Möglichkeit, Neutronenaktivierungen für wissenschaftliche Zwecke durchzuführen. Dies wird sowohl von wissenschaftlichen Arbeitsgruppen der JGU selbst, als auch von externen Gruppen anderer Universitäten oder Forschungseinrichtungen genutzt. Bestrahlungen sind dabei an verschiedenen Positionen (zentrales Bestrahlungsrohr, Karussell, Rohrpost, thermische Säule) möglich, welche sich im Neutronenfluss und im Neutronenspektrum unterscheiden. Dabei kann, je nach wissenschaftlicher Fragestellung, sehr individuell auf die Bedürfnisse der Experimentatoren eingegangen werden, was beispielsweise Bestrahlungsposition, -dauer, Reaktorleistung und anschließende Messungen angeht.

Abbildung 1: Blick in die Experimentierhalle des TRIGA Mainz (links) und in den Reaktorkern (rechts).

Die Flexibilität der NAA am TRIGA Mainz zeigt sich im breiten Spektrum an wissenschaftlichen Fragestellungen, die in den letzten Jahren bearbeitet wurden. Dies reicht von der Messung von Halogeniden in verschiedenen Mineralien-Proben für das Helmholtzzentrum Dresden-Rossendorf, über die Bestimmung des Uran-Gehalts in Quarzglas für IceCube [3] bis hin zur Untersuchung organischer Proben, wie die Bestimmung von Spurenelementen in Spinat für die Hochschule Geisenheim oder die Quantifizierung des NaCl-Gehalts in diversen Maus- und Humangewebeproben für die TU München [4, 5].

Neben klassischen Fragestellungen der NAA, wie Elementidentifikation und Gehaltsbestimmungen, kann Neutronenaktivierung auch für physikalische Grundlagenexperimente genutzt werden. So konnte beispielsweise die Intensität der 66.7 keV γ-Linie von Tm-171 mit hoher Präzision bestimmt werden [6], woraus wiederum der thermische (n,γ)-Wirkungsquerschnitt für dieses Nuklid, welches einen wichtigen Verzweigungspunkt im s-Prozess darstellt, ermittelt werden konnte [7]. Ebenso konnte durch die Herstellung einer hohen Aktivität (MBq Bereich) von Rb-88 ($T_{1/2}$ = 17.8 min) ein extrem schwacher Übergang bei 4742.5 keV beobachtet werden, aus welchem Rückschlüsse auf die Zwei-Phononen-Struktur dieses Zustands gezogen werden konnten [8].

Im Rahmen dieses Vortrags soll ein Überblick über die Möglichkeiten mittels Neutronenaktivierung am TRIGA Mainz und die damit ermöglichten wissenschaftlichen Experimente gegeben werden.

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Optimization of neutron activation analysis of rare-earth elements

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Instrumental neutron activation analysis (NAA) is a powerful tool for detecting trace elements within a wide variety of matrices samples. Additionally, NAA allows for multi-element determination of rare-earth elements (REE) within geological or archeological samples. Due to spectral interferences created by overlapping gamma photopeaks, high backgrounds due to Compton scattering, and uranium fission concentrations desired measurements can often be misinterpreted or buried in the continuum altogether. To enhance these procedures a database was created to help assess the likelihood that a certain lanthanide isotope and gamma ray is indeed the best one to be determined by NAA. First, the most common isotopes through the (n,γ) reaction of each element was determined, along with the strongest and most abundant gamma rays to be used, and a chart was formulated with their relevant properties. A weighting factor was constructed based on seven characteristics of the properties of the lanthanides: thermal cross section, resonance integral, natural abundance of the element itself, half-life of the activation product, the branching ratio, the I_{gamma}(relative) value, and relative detector efficiency of the photon spanning from 80 – 1596 keV. The seven individual characteristics were then added together and divided by the total possible value to get their total weighting factor. In addition, the application of gamma- gamma coincidence and Compton suppression was also evaluated to determine which radionuclides may be better determined.

Spectroscopy Database for Prompt Gamma Activation Analysis

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The first complete spectroscopy database to be used in Prompt Gamma Activation Analysis (PGAA) was published in 2004, i.e. it is more 20 years old [1]. During these years, it has been thoroughly tested and applied for ten thousands of successful analyses in the Budapest (BNC) and in Garching (MLZ) as well as a few other labs. This database contains the energy and partial gamma-ray production cross section values of the hundred strongest characteristic gamma lines appearing in the prompt gamma spectra for all naturally occurring elements.

Because of the large number of characteristic peaks in the prompt-gamma spectra, their in-lab evaluation became possible only in the 1990s. The earlier databases contained so many false peak identifications that they could not be used for reliable chemical analysis. Soon after the start of the PGAA lab at Budapest, it was realized that a systematic measurement campaign is necessary to determine the spectroscopic parameters of the detectable characteristic peaks. This first campaign took place between 1996 and 2003 first in the thermal neutron beam with the flux of 2.6×10^6 cm^{-2} s^{-1} then in 20-times stronger cold beam.

The library measurements strongly relied on the nuclear-physics experimental procedure used at Budapest based on the thorough calibration of the detection system, including broad-range energy calibration normalizing together the different sources [2], two-point energy calibration with correcting for the non-linearity [3]. Both were performed by Hypermet-PC [4], which was the only peak-analysis software running on Windows personal computers, also being capable of handling 16k spectra full of complicated peak overlaps. To this day, this program is still one of the best for such spectra, thanks to its peak-shape function and the built-in quality-assurance routines.

The measurement series was inspired by the establishment of the k_0 standardization method of NAA partly developed at the Budapest Research Reactor by András Simonits in the 1980s. Three spectra were acquired for each element: 1) a pure element spectrum of typically metal foils, oxide powders, 2) energy-calibration spectra based on chlorides or pure element measured together with PVC foils for determining the peak positions relative to the precisely known ^{36}Cl lines, 3) standardization spectra using simple stoichiometric compounds or water solutions to determine the partial cross section relative to the one of well-known values, e.g. H or Cl.

An automatic analysis procedure Prospero was developed in parallel [5] to determine the concentration values from the peak areas using statistical criteria and empirical qualifier numbers. Based on the experiences accumulated during the work with the database and the program, a few updates were released, e.g. with the determination of detection limits and new energy and cross-section values were introduced to correct for the discrepancies found in some analyses.

The database appeared in a few other releases, too, the PGAA database on the IAEA Nuclear Data Center [6] and the EGAF datafile [7] initiated by Richard B. Firestone.

After 20 years of successful application of the database, a need has arisen for its upgrade based on new measurements with the strong support of the IAEA. A measurement campaign was performed in Garching at the MLZ in its strong cold neutron beam between 2011 and 2020 to repeat and improve the old Budapest measurements with smaller sample masses and using freshly purchased chemicals. Approximately 80% of the measurements are complete. Unfortunately, it had to be suspended due to the stop of the FRM II reactor.

Besides the new, higher-quality spectra, new nuclear data became available, such as the new nuclear mass evaluations [8] yielding high-precision end-point energies for many elements redefining and further improving the energy calibration approach.

The re-evaluation has been going on since 2023 partly supported by the IAEA. As a result, the upgraded database will be available within a year and it is planned to be published in the Atomic Data Nuclear Data Tables. The talk presents the status of the project.

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[8] https://www-nds.iaea.org/amdc/

Machine Learning Methods for Prompt Gamma Activation Analysis

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Analysis of the spectra obtained with Prompt Gamma Activation Analysis (PGAA) is a well-developed method with relatively straightforward data treatment. However, due to the large number of gamma rays emitted after neutron capture, and the possible interference of the gamma rays from different elements, in the target material, this process can be time consuming. This is also additionally complicated by the fact that the peak shape in PGAA is influenced by several contributions, requiring careful analysis. Expert analysis can mitigate some of these problems, but there are situations when the analysis can get so complex that it is hard to achieve the necessary quality in identifying and quantifying the elements present in the target material in a reasonable time.

In the recent years, there is a large influx of Machine Learning methods applied to physics. They are used in various ways, for example to aid the analysis or control the measurement process. These methods are relatively easy to use, and versatile. For this reason, they were a natural choice for the PGAA. This research is currently being done in the framework of the BMBF funded collaborative project EvalSpek-ML (Grant number: 05D2022). The project encompasses the usage of ML methods in various fields of physics beside PGAA, such as astrophysics and neutron scattering. The overview of the project will be presented, but the main focus will be on the PGAA.

The research is focused on developing the ML method for the automated analysis of the PGAA spectra, but with the metrics that can compare this analysis with the expert one. Two kinds of solutions are being tested, one with the automated full analysis of the data, and the two-step one, where the first step of automation ends with the peak lists in the spectra with determined areas and errors, and the second one does the determination of the elemental composition of the sample. This would ensure that the results obtained with ML could always be compared to the results obtained by experts. This work is currently in progress, and only preliminary results will be presented.

One of the main issues in applying the ML methods for the PGAA spectra is the necessity for a large amount of data. Although data can be augmented with various methods to a degree, the number of the spectra available at the PGAA facilities is usually relatively small compared to the ones needed to ensure the successful training of the ML algorithms. The second problem with this is that the spectra measured are usually measured for a specific application or research, so the availability of spectra for different elements for the training is not evenly distributed. For this reason, the part of the research is focused on the introduction of the simulations of the PGAA spectra. This process is currently ongoing and preliminary simulation results will be presented.

The first batch of data was tested with multiple methods, including Linear Regression, Random Forest, 1D Convolutional Neural Network, Feed Forward Neural Network, and Autoencoder algorithms [1]. Some of the results can be seen in Figure 1, while the performance of the various approaches is described by metrics in Table 1.

Algorithm	**MSE**	**MAE**	**MSBE**	**RP**
Autoencoder	**0.38**	0.06	**1.42**	0.65
CNN	36.08	0.52	37.70	0.88
LR	72.84	1.64	75.94	0.82
FFNN	0.57	**0.05**	1.58	0.91
RF	61.05	1.32	63.38	**0.92**

Table 1: Metrics for the used algorithms. MSE and MAE represent Mean squared error and mean absolute error, respectively. MSBE and RP are new metrics introduced to reflect the specifics of the PGAA spectra. MSBE is a metric that penalizes the predictions outside of the uncertainty range, while RP is the average amount of elements that are predicted within their respective uncertainty range.

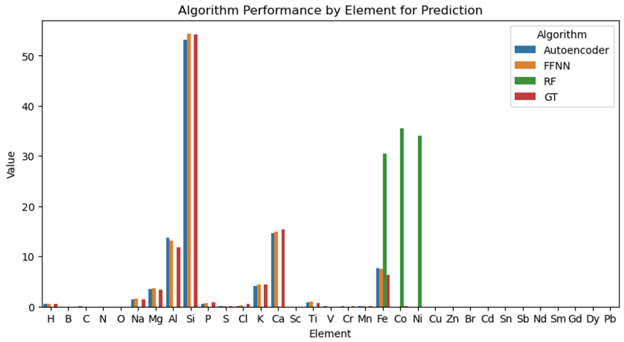

Figure 1: Average prediction.

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Neutron Activation Analysis with Full-Spectrum Least-Squares Fitting

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Typically, gamma spectra are analyzed by finding and fitting individual peaks with modified Gaussian shapes and then associating them with probable sources. Peak-search methods often have difficulties with resolving multiple overlapping peaks, especially in regions where multiple smaller peaks are masked by several overlapping larger peaks. Additional uncertainty in peak area can result from estimates of the local underlying continuum, especially in regions in which the continuum is obscured by many overlapping peaks.

Non-linear least-squares fitting methods [1,2,3,4] are often employed to deconvolve overlapping peaks. They are especially effective with unusual peak shapes, such as with the data from Supernova 1987A [5], and produce significant improvement in the results. However, these methods continue to have difficulties resolving multiple peaks which have greatly differing areas and an uncertain underlying continuum. In particular, analysis of weak sources with strong interference in complex spectra continues to be a difficult challenge with considerable uncertainties.

In an entirely different approach to analysis of gamma spectra, non-linear least-squares methods have been applied to the entire gamma specctrum in a program known as VRF (Visual Robust Fitting). With this approach, instead of finding and fitting individual peaks, the entire spectrum is fitted as a single, holistic, self-consistent set of data [6,7,8]. All of the peaks of each gamma source, and the continuum, are represented by a single, continuous curve that runs from the beginning to the end of the spectrum. X-rays and escape and summing peaks are included in each spectrum-wide curve. In this way, every count in every channel has some influence, however small, on the overall result. A single metric for the fit to the entire spectrum, $\chi2$, is minimized in a convergent iterative procedure until no further improvement is possible. For the VRF fitting procedure, $\chi2$ is the sum of squares of differences between the fit and the data, divided by the number of data points. Fitting all of the data in this manner directly results in full decomposition of multiple interfering peaks. This holistic method results in significantly better sensitivity in comparison with standard peak-search methods for analysis of weak gamma sources that are obscured by other sources [9,10].

The holistic method requires a fit to *all* peaks, no matter how small, so the VRF main library includes 2,367 radionuclides, decay x-rays, elemental x-rays for vacant K-shells, atomic masses, isotopic abundances, mass attenuation coefficients, common decay chains and nuclear reactions, all of which can be edited or added by the user.

To add prompt gamma neutron activation analysis (PGNAA) to VRF, the most recent database from the IAEA [11] was added to the main library. Code was added so that on startup all elements in the database appear in a selection list named *N-capture*. Code was also added to display analyzed elements in relative mass or as grams of mass in the irradiated volume if the neutron flux is known and uniform. A special shape for the peak of ^{10}B was created to fit its highly Doppler-shifted peak shape that results of the energetic reaction ^{10}B(n,α)^{7}Li. The VRF main library for prompt gammas from sodium was changed to include the decay of ^{23m}Na which, with a 20.2 ms half-life, can be considered as prompt. VRF was modified to read spectra with up to 65,536 channels. Finally, the VRF report was modified for analysis specific to PGNAA.

With these modifications in place, we proceeded with the main objective of this study, which is *to assess the agreement between the two quite different methods of PGNAA analysis -- individual peak-fitting and full-spectrum least-squares fitting.* We re-used prompt gamma spectra of five samples of concrete previously acquired at the Budapest reactor and analyzed [12] with Hyperlabs/ProSpeRo software [13]. The samples were prepared as part of a separate research project to investigate various levels of chloride in concrete [14]. These samples and their analysis provided an excellent opportunity for comparisons of individual peak-fitting and full-spectrum least-squares fitting under differing conditions. The results of these comparisons are presented.

Addition of the latest PGNAA database into the VRF main library is discussed in more detail. Differences between the database and the actual data are noted, suggesting possible further improvement to the database. Future work to understand the observed differences between the two methods of analysis and to add a capability to VRF to account for beam attenuation and self-absorption is planned.

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Prompt Gamma Analysis based on Inelastic Neutron Scattering

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The potential of the Prompt Gamma Analysis based on Inelastic Neutron Scattering (PGAINS) for non-destructive chemical analysis of large samples was already demonstrated several decades ago [1].

The FaNGaS (Fast Neutron-induced Gamma-ray Spectrometry) instrument, installed at Heinz Maier-Leibnitz Zentrum (MLZ) advances this analytical technique and makes it available for a broad community of industry and research [2].

PGAINS method principle

PGAINS method is based on the measurement of isotope-specific prompt gamma-rays emitted from a nucleus in an excited state after an inelastic interaction with a fast neutron, i.e. (n,n′γ) reaction, as schematically shown in Fig. 1.

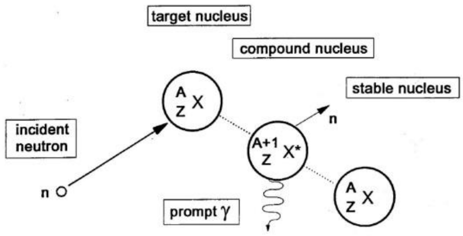

Figure 1: Schematic representation of the PGAINS method principle.

This prompt gamma radiation can be measured using high-resolution HPGe or medium resolution CdZnTe (CZT) detectors.

FaNGaS instrument & applications

The intense fission neutron beam delivered by the research reactor FRM II (Forschungs-Neutronenquelle Heinz Maier-Leibnitz) allows to investigate the fast-neutron induced prompt gamma-ray emission, offering new possibilities for the chemical analysis of large or small samples as a complementary method to conventional thermal- or cold-neutron based PGNAA.

The predominant reaction channel of fast neutrons at FaNGaS is the (n,n'γ) inelastic scattering reaction. Currently, the only one existing data catalogue of such reactions is the "Atlas of Gamma-rays from the Inelastic Scattering of Reactor Fast Neutrons", published in 1978 by Demidov et al. [3]. This data compilation is valuable and a relational database has been recently developed based on this Atlas [4]. However, it was yet never validated and further measurements of pure elements are required. Apart from building up a comprehensive catalogue of (n,n'γ) reactions another main objective is a continuous optimization of the instrument to improve the analytical sensitivity to tackle the industrial needs.

Among such important industrial applications is the characterization of rare-earth magnets in view of their recycling, following the EU Critical Raw Materials Act published in 2023 [4].

A numerical feasibility study was conducted for the most common type of industrial permanent magnets – FeNdB [5]. Two different measurement configurations were investigated using Monte Carlo simulations in PHITS v. 3.28 code – one with 50% efficient HPGe detector, the other one comprised of an array of 8 CZT detectors, 4x4 cm each, as shown in Fig. 2.

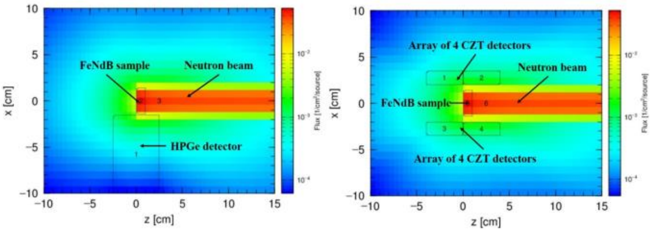

Figure 2: 2D view of the PHITS simulation models.

The spectra were collected with 16384 energy bins with the upper energy threshold set to 9000 keV. The number of primary neutrons is 10^{10} for each simulation corresponding to a neutron fluence of $1.62x10^9$ cm^{-2} at sample position

Results and conclusion

Measurements of the gamma-ray energy and partial cross-section were conducted for 22 elements. For these elements 1629 gamma-rays were observed, which correspond to 20% of the Demidov Atlas. Moreover, due to better energy resolution and measurement configuration 624 new gamma-rays were discovered. However, 155 gamma-rays were not observed, what can be attributed to interferences and lower mass of samples or due to the fact that some of these gamma lines were wrongly assigned. Further measurements are required to investigate this matter.

A rapid characterization of magnets requires a high neutron flux at sample position that cannot be achieved by using a portable neutron generator. Compact Accelerator based Neutron Source (CANS) based on a 5 MeV proton accelerator and a thick beryllium target for neutron production via the $^9B(p,xn)^9Be$ reaction allows to achieve a neutron emission 10^{10} s^{-1} towards the sample. An array of CZT detectors demonstrator an advantageous performance in terms of detection efficiency and measurement time.

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Actinide measurements with AMS and ILIAMS at VERA

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Accelerator Mass Spectrometry (AMS) is an ultra-sensitive method for detecting radionuclides independent emissions during radioactive decay. With AMS, anthropogenic actinides such as ^{236}U or ^{239,240,241}Pu released by human nuclear activity can be used as markers for environmental processes [1].

An AMS facility like the Vienna Environmental Research Accelerator (VERA) shown in Figure 1 consists of a source of negative ions (a) and two mass spectrometers (b, d) connected by a tandem accelerator (d). Stable isotopes can be measured in Faraday cups along the beamline and eliminating the molecular isobaric background via stripping in the accelerator enables the ultra-sensitive detection of long-lived radionuclides in single particle detectors, e.g. gas-filled ionization chambers (f). In the actinide beamline of VERA, a second high-energy analyzing magnet (e) drastically reduces tailing from neighboring masses even in this high mass range. This additional filter has enabled detection limits for ^{236}U/^{238}U below 10^{-14} [2] and unlocked the ^{233}U/^{236}U isotope ratio which could play an important role as a fingerprint for source term identification [3].

In AMS, detector counts must be normalized to either a stable isotope or a known quantity of an isotopic spike to produce reliable quantitative results.

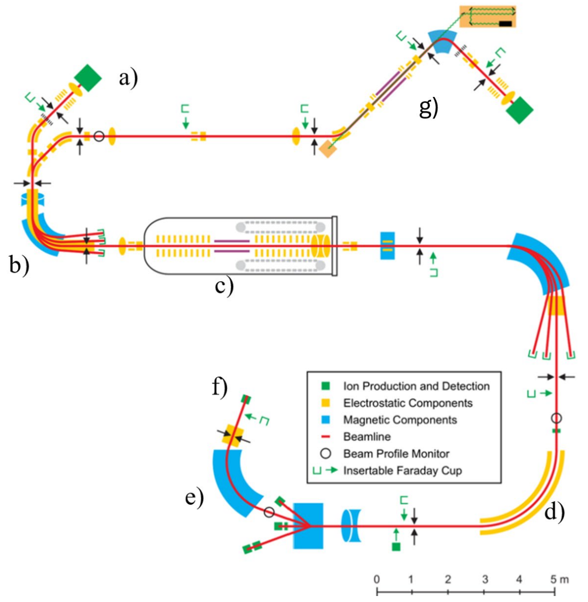

Figure 1: Schematic of the actinide beamline (a-f) and the ILIAMS setup (g) at VERA.

Beamtime at VERA is available to external users via projects such as RADIATE, ReMade@ARI or as a CORE facility of the University Vienna. A selection of recent actinide data will be presented.

AMS measurements were traditionally limited to cases where interfering atomic isobars either do not exist or do not form anions. For some lighter radionuclides interfering isobars that do form anions can be separated based on energy loss in matter due to differences in nuclear charge Z. As $\Delta Z/Z$ decreases towards heavier nuclei, the energy required for separation increases so that no AMS facility in the world was capable of separating isobars in the actinide range.

The Ion-Laser InterAction Mass Spectrometry (ILIAMS) setup at VERA (denoted by g) in Fig.1) combines an ion cooler with high-powered lasers or reactive gases to separate isobars via laser-photodetachment or selective chemical reactions before the ion beam is guided to VERA.

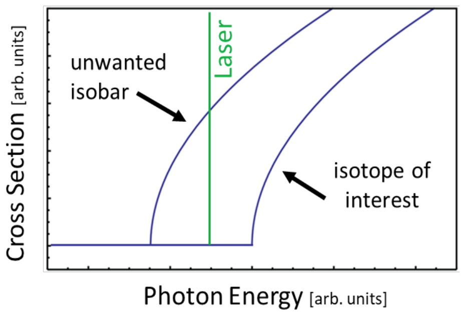

Figure 2: If the stability of the anion containing the unwanted isobar is lower than that of the isotope of interest, a laser with a photon energy in this gap can be used for selective photodetachment in ILIAMS

Recently, methods for the separation of U, Np and Pu isobars with ILIAMS were developed, making VERA the first and currently only AMS facility in the world capable of isobar separation in this high mass range.

These new capabilities were successfully employed in characterizing a prospective ^{236}Np spike material, where unwanted co-production of ^{236}U and ^{236}Pu is expected during the production process. Such a spike is urgently required for reliable normalization of measurements on environmetal ^{237}Np, the second most abundant anthropogenic actinide in the environment.

This work was supported by the Austrian Science Fund (FWF):[I-4803-N], RADIATE [Horizon2020 grant agreement No 824096] and a Dimitrov Fellowship of the Austrian Academy of Sciences. The authors wish to thank all colleagues who have provided samples for actinide analysis at VERA.

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Determination of circular economy related elements in LED light sources

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Nowadays, the presence of waste electrical and electronic equipment (e-waste) is one of the main environmental problems [1]. By recycling them, their impact on the environment could be significantly reduced. Furthermore, they could be a valuable secondary source of raw materials for industry, especially in the case of valuable elements and critical elements [2], which are elements that are both of high importance for the economy of a given region and have a high supply risk in that region. In the case of the European Union, such elements for example include gallium, antimony, tungsten and all rare earth elements (REEs) [3].

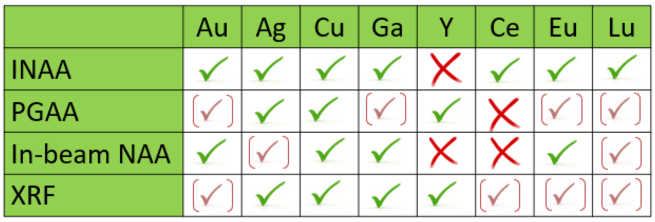
Some e-waste can contain notably high concentrations of critical elements such as LED chips from LED light sources. These are typically made using semiconductor diodes containing gallium [4] and phosphors containing various REEs [5]. In our work, we have investigated the feasibility of determining the comprehensive elemental composition of various LED chips covering all critical, valuable and hazardous elements using a combination of different neutron-based analytical methods. We have also examined - with the help of the results of neutron-based methods - the potential of hand-held X-ray fluorescence spectroscopy (Hh-XRF), which is a faster method than neutron-based techniques and can even be adapted for industrial applications.

For the measurements, the LED light sources were disassembled, and the LED chips were removed. The LED chips were measured with INAA at the research reactor of the Budapest Neutron Centre with both short and long irradiation. For quantitative analysis, the k_0 method was used. The samples were also analysed with PGNAA, supplemented by in-beam NAA measurements of the activated samples during the PGNAA measurement to improve the detection limits of elements that are better measurable from the decay spectra compared to the prompt spectra.

The LED chips were further measured with a Bruker Tracer 5g Hh-XRF spectrometer. For this technique, qualitative and quantitative analysis is typically performed using a matrix matched calibration method provided by the manufacturer, however this has not been available for LED chips. Due to the lack of a proper calibration mode, the peaks identified in the XRF spectra were used for the qualitative analysis, and to assess the feasibility of quantitative analysis, the peak areas of the characteristic lines of each element were compared with the results of neutron-based methods for different samples.

The INAA method was able to quantify several valuable, critical and hazardous elements in LED chips, e.g. Au, Ag, Br and various REEs, and for most of the important elements low detection limits were achieved. However, yttrium, which is a presumed component of LED chips and a key critical element, was not measurable with this technique and the obtained detection limit was outstandingly high. However, by applying the PGNAA method, it was possible to quantify this element. In addition, this technique was also suitable for the determination of other crucial elements such as Cu and Ag, and in combination with in-beam NAA it was also capable of the measurement of further key elements such as Au and Ga. However, the detection limits achieved with the neutron beam based methods proved insufficient for the detection of some other important elements, such as some other REEs successfully quantified with INAA. It can be concluded that the combination of the different methods is crucial for the comprehensive analysis of the LED chips.

In general, Hh-XRF achieved worse detection limits than neutron-based methods, and the presence of some important elements (e.g. Au and some REEs) was only measurable in a subset of the LED chips containing higher concentrations of the investigated element. However other key elements such as Ga, Y and Ag were detectable in all LED chips using this method. The measurable elements for each method are summarised in Figure 1, focusing on the most important critical or valuable elements. The peak areas measured by Hh-XRF were only in limited correlation with the concentrations measured by neutron-based methods. The two identified main reasons for this are the inhomogeneity of small size LED chips and the significantly different matrix composition of some LED chips, which significantly influence the results obtained with Hh-XRF. Which have to be eliminated or considered in the future to implement quantitative analysis with Hh-XRF.



	Au	Ag	Cu	Ga	Y	Ce	Eu	Lu
INAA	✓	✓	✓	✓	✗	✓	✓	✓
PGAA	(✓)	✓	✓	(✓)	✓	✗	(✓)	(✓)
In-beam NAA	✓	(✓)	✓	✓	✗	✗	✓	(✓)
XRF	(✓)	✓	✓	✓	✓	(✓)	(✓)	(✓)

Figure 1: Applicability of each method for the measurement of the most important critical and valuable elements in LED chips

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Determining chlorine in solids at ppb-levels and below using neutron activation and accelerator mass spectrometry

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Neutron activation analysis using decay counting of the activated element is a well-established method in elemental analysis. However, for chlorine there is a more effective alternative to measuring the decay of the short-lived activation product chlorine-38 ($t_{1/2} = 37.24$ minutes) – accelerator mass spectrometry (AMS) of ^{36}Cl ($t_{1/2} = 301$ ka). The relatively high neutron capture cross section of chlorine-35 for thermal neutrons (43.7 b) combined with the efficiency of the AMS technique for chlorine-36 allow for determination of chlorine down to ppb-levels using practical samples sizes and standard exposure durations. The combination of neutron activation and AMS can be employed for a few other elements (nitrogen, thorium, and uranium) as well.

For bulk solid samples an important advantage of the method is that laboratory contamination can be rendered irrelevant, since the ^{35}Cl in the sample is activated to ^{36}Cl, and surface chlorine can be removed after the irradiation. Unlike the sample chlorine, subsequent contamination incurred during sample preparation will not carry a prominent chlorine-36 signature. After sample dissolution and addition of sufficient amounts of chlorine carrier, the produced chlorine-36 and thus the original chlorine-35 of the sample can be determined using AMS.

We have developed and applied the method for the analysis of chlorine in steel samples. The chlorine content of steel is of interest to nuclear industry, precisely because of above mentioned high neutron capture cross section for chlorine-35, which leads to accumulation of chlorine-36 as long-term nuclear waste. We carried out irradiation of the samples at the TRIGA Mk. II reactor of the TRIGA Center Atominstitut in Vienna and ^{36}Cl-AMS measurements at the Vienna Environmental Research Accelerator.

Materials analysis using MeV ions: Fundamentals and applications

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Ion-beam analytical (IBA) techniques use a beam of energetic ions, provided by a medium-energy particle accelerator, to induce atomic/nuclear interactions, and detect these interactions in order to get insight into the elemental and structural composition of a sample. Several types of interaction products can be detected, which results in a variety of ion-beam based analytical methods. All of them have their own advantages compared to others and to non-IBA techniques, and, in addition, some of them can be used simultaneously to get synergistic information of the sample. When combined with a scanning nuclear microprobe, the obtained information can be displayed on element maps with spatial resolutions in the micrometer range. IBA techniques are widely used in various fields of science and technology, for instance, in materials science, forensics, life sciences, archaeology, etc.

The presentation will provide an introduction into the basics of a few of the most commonly used IBA techniques, including the necessary instrumentation and the underlying physical phenomena. Particular attention will be paid to i) Particle Induced X-ray Emission (PIXE) analysis, ii) Rutherford Backscattering Spectrometry (RBS) and iii) Time-of-Flight Elastic Recoil Detection Analysis (ToF-ERDA). The capabilities of these selected techniques will be shown via case studies. For all of them, the case of a rather standard, routine measurement will highlight the strength of the given IBA technique. In addition, a non-trivial case, where methodological developments were necessary to extract new kinds of information from the measurements, will point out the potential of IBA.

Particle Induced X-ray Emission

In Particle Induced X-ray Emission (PIXE) analysis, characteristic X-rays induced by the impinging particle beam are detected. PIXE is trace-element sensitive, providing detection limits potentially down to the ppb range.

In a recent project, we have developed a fully automated measurement system, based on the scanning nuclear microprobe in the Tandem Laboratory at Uppsala University, that is capable of detecting micrometer-sized grains in samples with area up several cm^2 by micro-PIXE, in a time-efficient manner [1]. The presentation will summarize the work, including the performance tests for the grain-detection efficiency, and measurement results obtained from peri-implant soft tissue sections containing metal microparticles that originate from dental implants (see Fig. 1).

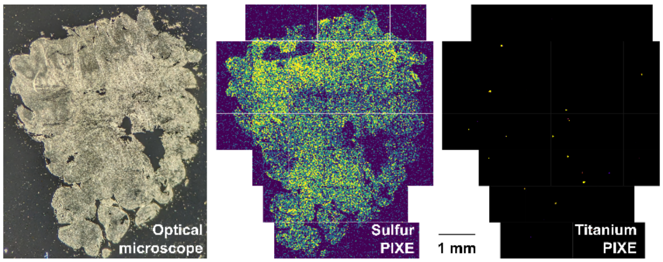

Figure 1: Optical microscope image, sulfur and titanium PIXE maps of a peri-implant soft tissue section. The S map is used for easy positioning. Several micrometer-sized grains are visible in the Ti map.

Rutherford Backscattering Spectrometry

In Rutherford Backscattering Spectrometry (RBS), the detected interaction products are the projectile particles that get elastically backscattered from the target. These particles carry information on the mass of the backscattering target atom, as well as its depth in the matrix, resulting in a method that is highly depth-sensitive.

In a recent work, we applied RBS to investigate the transport process of post-synthetically introduced catalytic sites in metal-organic framework (MOF) single crystals (see Fig. 2). First, we developed a method that quantifies the elemental depth gradients of these active sites in the sample [2]. When considering the transport as Fickian diffusion, the diffusion coefficients are directly obtained from the depth gradients. Then, we applied the method to the systematic investigation of UiO-67-type MOFs. We revealed that by introducing a mixture of different linker molecules into the crystals, the diffusion speed can be tuned, and consequently, the spatial distribution of the catalytic sites in the MOFs can be optimized [3].

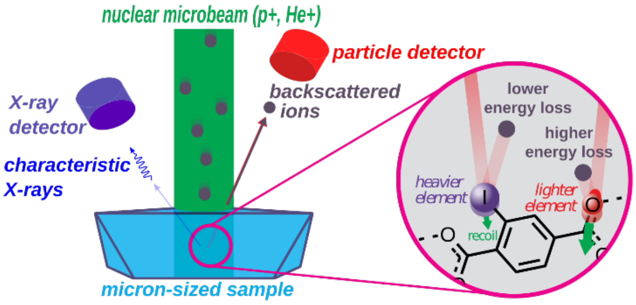

Figure 2: Schematic drawing of elemental depth-profiling of MOF single crystals.

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Validation of MCNP6 and FISPACT calculations to predict the activation of radioisotopes used for medical purposes

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In recent years the European Union invested great effort to assess the operational research reactors, their overall (even financial) conditions and available capacities [1]. Among the several applications of a research reactor medical isotope production is of great importance. The aim of the SECURE (Strengthening the European Chain of sUpply for next generation medical RadionuclidEs) project is to make a major contribution to the sustainability of medical isotope production and its safe application in Europe [2] coordinated by POLATOM [3]. This includes target development, and reliable solutions for production methods of therapeutic and diagnostic alpha and beta emitter radionuclides also in research reactors.

This presentation is limited only to show the feasibility calculations of producing beta-emitting radionuclides via MCNP6 and FISPACT-II software. At first, the flux characterization of three highly different vertical irradiation channels of the VVR-type 10-MW Budapest Research Reactor (BRR) was carried out. The resulting MCNP6 F4 tally output data were tabulated according to the CCFE-709 group energy structure, one of the native energy-bin sets of FISPACT-II, ranging from 10^{-5} eV to 10^{9} eV. Using the neutron field parameters and the composition of the target, a radioisotope inventory can be obtained using the FISPACT-II code, for any time instant during and after the irradiation.

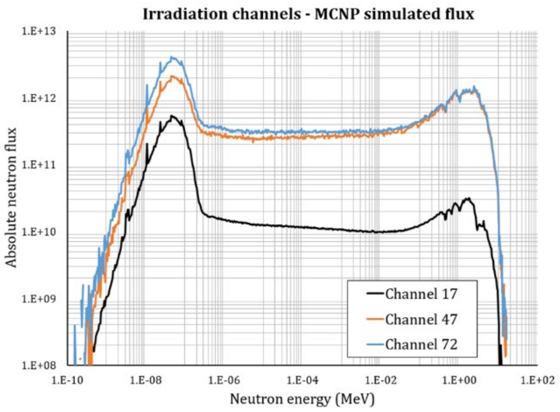

Figure 1: Visualization of the neutron energy distribution based on the MCNP F4 tally output data.

We completed the activity calculation of the following four nuclides used in radiotherapy, namely ^{177}Lu which currently forms the basis of the majority of the radiopharmaceuticals used for therapy in clinical trials [4]; ^{161}Tb, an emerging, potential replacement for ^{177}Lu due to its advantageous decay properties [4,5]; and two additional promising beta emitters in tumour treatment, ^{111}Ag [6] and ^{199}Au [7].

Gd, Pd, Pt, and Lu-containing samples of natural isotopic composition were irradiated in the three selected vertical channels of the BRR to produce ^{161}Tb, ^{111}Ag, ^{199}Au and ^{177}Lu, respectively. The experimental activities of the irradiated samples were measured using well-calibrated HPGe detectors placed inside low-background counting chambers. The results were compared with the FISPACT-II calculations, and an agreement within 30% was found. Only few discrepant cases were identified which require further investigation. The method can be generalized to assess the yield for any yet untested irradiation site.

Acknowledgement

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Szilárd-Chalmers investigations of neutron-irradiated gold foils

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In 1934 L. Szilárd and T. A. Chalmers discovered changes in the chemical environment in the course of nuclear reactions. In an (n,γ) reaction, the resulting recoil energy from prompt gamma emission is big enough to break up the chemical bond between the activated atom and its residual molecule [1]. Over the course of conducting numerous experiments the Szilárd-Chalmers effect has been examined concerning the creation of the radioactive isotope Au-198 via the ^{197}Au(n,γ)^{198}Au nuclear reaction [2]. Various experiments involving gold foils have been tested to study in depth the effect and possible side-reactions taking place when the gold foil is place in a liquid during activation. The experiments were carried out using the TRIGA Mark II reactor at the TRIGA Center Atominstitut [2].

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Investigation of the ^{99}Mo production via neutron capture ^{98}Mo(n,γ)^{99}Mo with a high-current accelerator-based neutron source

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^{99}Mo serves as the precursor to ^{99m}Tc, an essential radioisotope used in approximately 80% of all nuclear medicine procedures. The demand for ^{99m}Tc, used in medical imaging, is steadily increasing [1]. ^{99}Mo is primarily produced through fission of ^{235}U in high flux reactors [2]. The supply chain disruptions associated with aging reactors facing potential shutdowns and radioactive waste management emphasize the crucial need for alternative production methods, particularly in regions like Germany, where demand remains high [3].

Alternative approaches to ^{99}Mo production are based on neutron capture reactions, fast neutron capture reactions, and photo-neutron reactions by irradiation of natural composition or ^{98}Mo enriched molybdenum samples. Reduced radioactive waste and simplified processing schemes are advantages of these methods compared to ^{235}U-fission path.

Accelerator-based technologies present a promising solution to ensure the supply of essential medical isotopes while directly addressing concerns related to radioactive waste management. Utilizing a high-current accelerator-based neutron source (HiCANS), as proposed in the ambitious HBS (Jülich High Brilliance Source) project [4], presents such an alternative approach to ^{99}Mo production. The ^{99}Mo production via a HiCANS involves (n, γ) reaction from fast neutrons generated by 70 MeV protons interacting with a heavy metal target, moderated and reflected in a Target-Moderator-Reflector (TMR) unit.

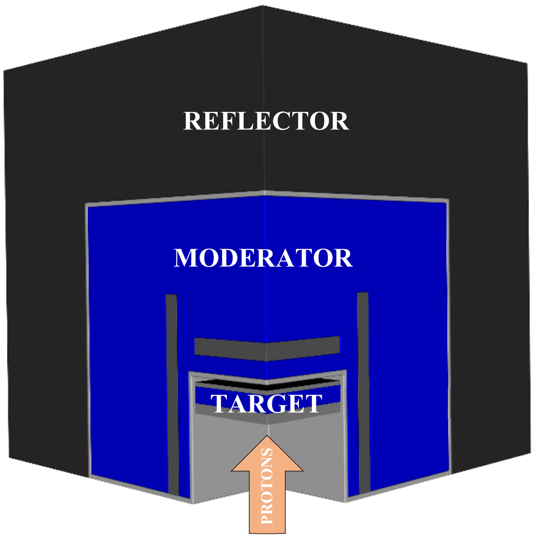

Figure 1. The target-moderator-reflector unit containing the molybdenum plates positioned around the target.

Monte Carlo simulations, utilizing PHITS code, are performed to examine various parameters, including target size and proton beam currents while limiting the target power density to 3 kW/cm^2.

Our study assesses the effects of multiple parameters such as TMR dimensions, materials, and compositions on the activation of a natural Mo plate for the production of ^{99}Mo.

This work provides important insight into the feasibility and development of ^{99}Mo production through accelerator-based neutron sources, establishing the foundation for future advancements in medical radioisotope production technology.

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EducTUM: Eine interaktive Plattform für die Aus- und Weiterbildung sowie den Kompetenzerhalt im Bereich der zerstörungsfreien Analyse von radioaktivem Material aus Stilllegung und Rückbau kerntechnischer Anlagen

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Die Stilllegung kerntechnischer Anlagen stellt große Herausforderungen an die Deklaration der hierbei entstehenden Abfälle. Dies wird durch Änderungen bestehender bzw. Einführung neuer Regelungen, auch im Hinblick auf bereits existierende (Alt-)Abfälle, weiter verschärft.

Da sich der Rückbau noch über Jahrzehnte hinziehen wird, erfordert dies den langfristigen Erhalt der entsprechenden spezifischen Fachkompetenzen. Dies gelingt nur durch kontinuierliche Fort- und Weiterbildung des bereits involvierten Personenkreises bei gleichzeitiger Ausbildung des technisch-wissenschaftlichen Nachwuchses. Auch die allgemeine Information der Bevölkerung darf hierbei nicht außer Acht gelassen werden.

Das vom BMBF geförderte Vorhaben EducTUM (Förderkennzeichen 15S9443) hat die Aus-, Fort- und Weiterbildung, den Kompetenzerhalt sowie die Vermittlung von allgemeinen Informationen aus den Bereichen der Charakterisierung radioaktiver Abfälle und der Produktkontrolle zum Ziel. Es adressiert einen breiten Personenkreis, von allgemein an der Thematik interessierten Personen, über Anwender bis hin zu Fachexperten. Die Art der Wissensvermittlung erfolgt deshalb entsprechend dem Kenntnisstand der einzelnen Personengruppen.

Der Ausgangspunkt hierfür ist die Webplattform www.eductum.de. In der aktuell stattfindenden ersten Phase ihrer Entwicklung erfolgt eine Beschränkung auf Bereiche der zerstörungsfreien messtechnischen Untersuchungen an radioaktiven Abfallgebinden und hier im speziellen auf Messverfahren, welche Gamma- und Röntgen-Strahlen nutzen.

Die Besonderheit an EducTUM ist die Möglichkeit professionelle Programme online zur Vertiefung der vermittelten Inhalte zu nutzen und der damit verbundene direkte Bezug in die Praxis. Ein Beispiel hierfür ist das Gamma-Spektrometrieprogramm LVis, mit dem online Messungen mit unterschiedlichen Detektoren an realen Proben möglich sind. Die Proben stehen an einem Messplatz in der Radiochemie München RCM und sollen in unregelmäßigen Zeiträumen getauscht werden.

Die grundlegenden Ideen und Zielsetzungen von EducTUM werden aufgezeigt und der aktuelle Stand der Umsetzung vorgestellt. Es wird auf die Nutzung von EducTUM für die Durchführung von online-Gamma-Spektrometrie-Praktika eingegangen.

^{89}Zr-PET-Imaging of Murine Colorectal Cancer Models using Site-Selectively Functionalized Huma Serum Albumin

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Human serum albumin (HSA) has been investigated as a drug-carrier system in cancer therapy due to its long plasma half-life and enhanced uptake in malignant tumor tissue.[1,2] However, there is a lack of clinical imaging strategies to stratify patients who would benefit from albumin-binding drugs. In this study, we radiolabeled HSA with zirconium-89, a positron-emitting radionuclide, with the aim of providing an imaging tool to study its accumulation in tumor tissue over time, particularly at late time-points post injection (p.i.) of the radiotracer.

Methods

The novel radiotracer [^{89}Zr]Zr-DFO*malHSA was prepared by bioconjugation of the bifunctionalized chelator and subsequent radiolabeling. Afterwards, it was analytically characterized, and evaluated *in vitro*. Subsequently, [^{89}Zr]Zr-DFO*malHSA was administered to Balb/c mice bearing subcutaneous or intraperitoneal CT26 tumors, and tumor uptake was assessed over 72 hours by PET/CT and *ex vivo* biodistribution. The stability of the radiotracer was assessed *in vitro* and *ex vivo*.

Results

[^{89}Zr]Zr-DFO*malHSA was obtained in high radiochemical yield (RCY > 70%) and purity (RCP > 99%). *In vitro* stability in both saline and human blood serum was greater than 90% for 7 days. In accordance with previously published literature, no significant cancer cell uptake could be observed *in vitro*.[3] After 30 minutes p.i., most of the radioactivity was detected in the blood pool (30 ± 4 %ID/g). [^{89}Zr]Zr-DFO*malHSA accumulated in the tumor tissue within 24 hours (9 ± 2 % D/g), and tumor uptake did not change significantly up to 72 hours (11 ± 3 %ID/g at 48h p.i. and 8 ± 1 %ID/g at 72h p.i.). The tumor/blood ratio improved over time, tumor/kidney and tumor/liver ratios remained constant. The *ex vivo* stability of the radiotracer in mouse serum showed >99% in the mouse serum.

Conclusion

A site-selective radiolabeling approach of HSA using zirconium-89 for PET-imaging *via* DFO* was successfully developed. [^{89}Zr]Zr-DFO*malHSA demonstrated excellent *in vitro* and *ex vivo* stability. *In vivo* experiments showed high tumor uptake with favorable tumor/background ratios in mice bearing CT26 allograft tumors. [^{89}Zr]Zr-DFO*malHSA shows great potential for future applications to further investigate albumin homeostasis in cancer and to stratify patients who would benefit from albumin-based therapies.

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Investigating the activation of a NPP concrete bioshield

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An ever-growing number of nuclear power plants are aging or in decommissioning, representing considerable amounts of structural waste. Most of it is inactive, and sorting this waste thoroughly is crucial to minimize the material classified as radioactive [1]. For this, a reliable inventory of the activation in different structural elements is necessary, and made time and cost-efficient by combining targeted sampling and experimental measurements with numerical simulations [2].

In this study, depth profiles of activation in the concrete biological shield of the nuclear power plant Greifswald Unit 2 were developed and used to refine Monte-Carlo calculations. To better apprehend the complexity of activation in the concrete, spatially resolved activation was investigated with a combination of autoradiography and Raman microscopy.

Depth profiles of activation

The concrete biological shield is the structural element most prone to activation, with its proximity to the reactor pressure vessel (RPV). Cores were drilled at strategic positions in the concrete bioshield and the main gamma-emitters ^{60}Co, ^{152}Eu and ^{154}Eu were analyzed with gamma spectrometry. The unrestricted clearance of the concrete near the RPV is limited by high levels of ^{152}Eu. The activation depth profiles suggest that the gamma-emitters are produced by thermal neutron capture [3]. Our calculation results are in very good agreement with the experimental values and follow the same trends, making the model developed for the calculation robust (see Figure 1).

The activity distribution can be reliably predicted in the rest of the biological shield for the gamma-emitters without further sampling.

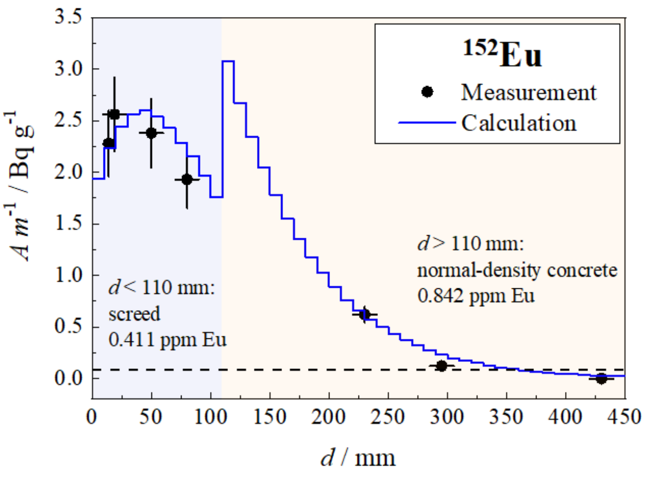

Figure 1: Measured and calculated depth profile in one drill core in the concrete bioshield for ^{152}Eu, the dashed line represents the unrestricted clearance level according to German regulation [4].

Spatial distribution of activity

Because concrete is a complex matrix, a better understanding of its activation can tremendously help with constructing and refining more reliable computational models, and to simplify the experimental work needed for accurate predictions. The spatial distribution of activation across the different mineral phases in concrete is not yet well understood. In this study, concrete drill cores were carefully marked and photographed, followed by autoradiography imaging of their surfaces. Hotspots were identified, and the corresponding mineral phases were determined using Raman microscopy.

The feldspars were the predominantly activated mineral phases. The results were consistent at different depths in the concrete bioshield, proving the reliability of the measurements.

Conclusions

By combining carefully selected experimental methods with numerical simulations, the tasks of sorting the structural materials and releasing the non-active waste can be achieved faster and cost-effectively. This approach can also reduce the occupancy rate within repositories for nuclear waste.

The investigation of mineral phase specific activation enables fast initial predictions of the overall activation of concrete for future decommissioning projects. Using only a powder X-ray diffraction (XRD) measurement, valuable insights can be gained before conducting more time-consuming and labor-intensive radioanalytical methods.

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Development of Radiochemical Separation Techniques for AMS Measurements of Actinides

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Accelerator Mass Spectrometry (AMS) is an ultra-trace analytical technique capable of measuring radionuclides down to ppq levels (i. e. fg/g) and even below. Thus, AMS is especially competitive to classical radioanalytical techniques, such as α, or γ-spectrometry, for nuclides with half-lives > 10 years.

Amongst these radionuclides, actinides are easy-to-measure AMS nuclides as most of them lack isobaric nuclides with similar long half-lives. Hence, the measurement of actinides using AMS possesses a broad range of applications including nuclear safeguards and forensics[1], environmental studies[2], astrophysics[3] and nuclear cross sections measurements[4] to name just a few.

The new 1-MV AMS facility HAMSTER (Helmholtz Accelerator Mass Spectrometer Tracing Environmental Radionuclides) at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Germany, will be especially dedicated to the analysis of actinides in ultra-trace levels. In addition to a multi-cathode cesium sputter ion source a state-of-the-art laser ion cooler setup will be installed at HAMSTER to access a broader range of radionuclides by enhanced isobar suppression.

Figure 1: Laser ion cooler setup at the HAMSTER facility.

The HAMSTER facility is also equipped with two new chemistry labs with capabilities for chemical sample workup for a broad range of radionuclides, especially actinides. These labs are operating under quasi-clean room conditions and are equipped with dedicated fume hoods for working with hydrofluoric acid and flow benches to minimize sample contamination on the ultra-trace level. Various radiochemical separation schemes for actinides (i.e. U, Pu) and other radionuclides are in routine operation[5,6] whilst the separation of other actinides (Pa, Np, Am/Cm) is currently under development. In this presentation, different radiochemical work-up procedures for various environmental matrices (see Figure 2) will be discussed and the future capabilities of the HAMSTER facility are highlighted.

Figure 2: Compilation of different sample matrices for actinide analysis.

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Electron macroscopy: Enhancing autoradiography by utilizing β^--sources

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Autoradiography is a commonly used imaging technique for radioactive samples for widespread applications. The use of radiosensitive imaging plates in combination with a phosphorimager allows for high sensitivity and high spatial resolution. Since the autoradiographic image does not necessarily contain information of the shape or placement of the imaged object on the imaging plate, the alignment of the image with the physical object often remains a challenge.

Here we report the use of a portable beta minus emitter as a simple and fast way to highlight the edges of a sample, obtaining direct localization information regarding the dimensions and shape of the sample. Thereby it is possible to get a combined image that contains both the autoradiographic information and the physical shape of the sample.

Experimental

Imaging was performed using a commercially available imaging system (HD-CR 35 NDT, DÜRR NDT). A sealed Sr/Y-90 source (220 kBq) with a pinhole was used as a β^--emitter and placed 15 cm above the imaging plate to achieve even exposure. This irradiation was typically done in the last 10 minutes of autoradiographic exposure.

A small calcium fluoride disc (d=2.54 cm, h=0.5 cm) which was implanted with radioisotopes [1], was investigated using the above-mentioned method.

Additionally a small selection of different items were imaged, to show the influence of different materials (aluminium, paper, PMMA,…) and thickness of the samples.

Results & Conclusion

After short irradiation time with a β^--emitter during autoradiographic exposure both the region of interest and the shape of the calcium fluoride disc could be visualized in one image (Figure 1). Therefore, the area implanted with radioisotopes could be successfully localized on the sample itself. This approach is especially useful when adequate x-ray sources are not available, and the investigated sample sufficiently absorbs radiation from the source. The effect of different materials and their thickness will also be discussed.

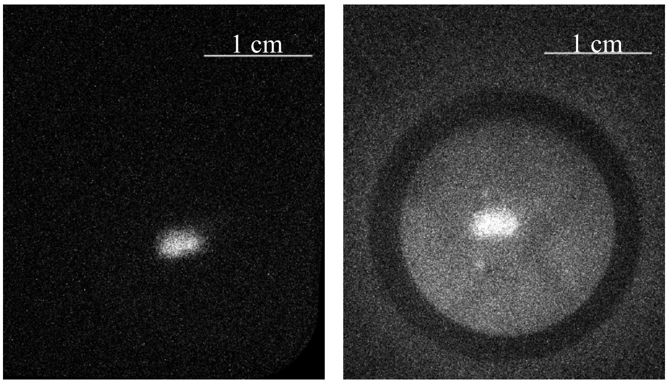

Figure 1: Two autoradiographic images of a disc-shaped sample implanted with radioisotopes[1]. The left image is a regular autoradiographic image. In the right image the shape of the sample was made visible using a Sr/Y-90 source.

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Practical applications of neutron activation analysis and γ-spectroscopy in medical isotope production research

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The application of medical isotopes for diagnostic and therapeutic applications is increasing due to recent developments in imaging technologies (eg total-body positron emission tomography – TB-PET) and the outstanding success in radiotherapy with alpha-emitters. The world-wide production of medical isotopes struggles to keep up with the demand due to aged production sites or limited availability of suitable target material. This sparks interest exploring alternative production routes and non-standard medical isotopes to circumvent potential future shortages.[1]

Radionuclidic purity is one major quality criteria in radiopharmaceutical production as long-lived radioisotopes would accumulate within the body. This leads to unnecessary increased dose for the patient and reduced image quality.[2] The power of neutron activation analysis and γ-spectroscopy is often underestimated when it comes to assess target purity and trace metal content. We selected three isotopes (see Table 1) where we applied neutron activation analysis (NAA) and/or γ-spectroscopy to either assess the target material, separation efficacy or final product.

isotope	$t_{1/2}$	decay	production	use
^{64}Cu	12.7 h	β^-, β^+	^{64}Ni(p,n)^{64}Cu ^{63}Cu(n,γ)^{649}Cu	diagnosis
^{199}Au	3.1 d	β^-	^{197}Au(n,γ)^{198}Au(n,γ)^{199}Au ^{198}Pt(n,γ)^{199}Pt$\rightarrow$^{199}Au	diagnosis therapy
^{225}Ac	10 d	σ	^{229}Th/^{225}Ac generator	therapy

Table 1: Selected isotopes discussed with their physical properties, current production route and clinical application [3,4, 5, 6]

Copper-64

Copper-64 is routinely produced via ^{64}Ni(p,n)^{64}Cu in a cyclotron, but requires solid targets not widely abundant.[3] Alternatively, it can be produced via neutron capture of ^{63}Cu giving ^{64}Cu in low molar activity.[4] Using the recoil upon neutron capture, the molar activity can be increased if the target material has a high stability, such as phthalocyanines. Copper(II)phthalocyanines can be easily produced but require a molybdenum catalyst. In order to obtain pure 64Cu upon neutron irradiation, complete removal of molybdenum in the target material is important. We assessed the purity of our target material via NAA confirming the absence Mo indicated by the absence of the 140 keV line from ^{99}Mo/^{99m}Tc.

Gold-199

Gold-199 is a potential radioisotope for theranostic application and is mostly produced from ^{197}Au leading to low molar activities and a mix of ^{198}Au and ^{199}Au. Alternatively, it can be produced from ^{198}Pt(n,γ)^{199}Pt$\rightarrow$^{199}Au requiring efficient separation of gold from platinum.[5] Separation efficiency for different extraction agents was determined by γ-spectroscopy. Although high purity Pt-wire was used for the production, γ-spectroscopy revealed iridium impurities indicated by the emission at 316 keV from Ir-192.

Actinium-225

Actinium-225 is an alpha-emitter undergoing four alpha emissions making it very potent for therapy.[6] Due to the absence of suitable γ-emission, direct measurement of ^{225}Ac is only feasible with α-spectroscopy. Sample preparation for α-spectroscopy is elaborate and α-spectrometer are not routinely available. Alternatively, the γ-emissions of the decay products ^{221}Fr and ^{213}Bi with their emissions at 219 keV and 440 keV, respectively, allows the use of γ-spectroscopy after sufficient equilibration time. We compared the radionuclidic purity of ^{225}Ac from an in-house developed ^{229}Th/^{225}Ac generator using α- and γ-spectroscopy. We obtained similar results for both methods showing the use of more available γ-spectroscopy as suitable for determination of radionuclidic purity of the α-emitting ^{225}Ac using daughter isotopes.

Conclusion

Neutron activation analysis is not often employed in medical isotope production research. Herein, we presented its application with the example of ^{64}Cu and ^{199}Au to obtain valuable information about target purity and separation efficiency. Additionally, we showed the indirect analysis of α-emitters using γ-emissions from its daughters on the example of ^{225}Ac.

In summary, both NAA and γ-spectroscopy should be more utilised in medical isotope production research, especially to determine target purity and radionucllidic purity. Furthermore, its application could also be extended towards investigation of trans-metallation in radiometal labelling.

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Using bovine bones as a toll for monitoring ^{90}Sr release in the environment

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In nuclear fission, fission products are produced, many of which are radioactive. The release of these radionuclides is viewed with concern by the public.

In the course of the Fukushima Daiichi nuclear accident (2011), large activities of radionuclides were released into the environment. While other radionuclides (e.g., ^{134}Cs, ^{137}Cs, ^{131}I, etc.) are not only volatile but also allow straightforward detection and quantification, ^{90}Sr has been emitted in lower quantities, and is notorious for its laborious sample preparation. Data on ^{90}Sr from Fukushima are, therefore, scarce by comparison. With its chemical similarity to calcium and its relatively long half-life of 29 years, it poses a potential threat to human and environmental health. Due to its chemical properties, radiostrontium is known to accumulate in bone tissue.

Previous studies have shown that, if any, increased environmental levels of ^{90}Sr are to be found in close vicinity to the Fukushima Daiichi reactors [1]. An interesting research opportunity arose when bones from a deceased cow that had lived approximately two kilometers from the reactor and was roaming freely for around one year after the accident were obtained. The ^{90}Sr content in these bones was investigated by LSC after its separation, which may be an important piece of the strontium release after a nuclear accident puzzle.

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