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HORIZON 2020

HORIZON 2020 RESEARCH AND INNOVATION FRAMEWORK PROGRAMME OF THE EUROPEAN ATOMIC ENERGY COMMUNITY

Nuclear Fission and Radiation Protection 2018 (NFRP-2018-4)

Project acronym: **SANDA**

Project full title: **Solving Challenges in Nuclear Data for the Safety of European Nuclear facilities**

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Editorial

Dear participants of the SANDA workshop on actinide target preparation and characterization – the need for radioanalytical chemistry,

I welcome you at the JRC Geel site. One of the laboratories of the Joint Research Centre of the European Commission. I contacted you all to participate in this SANDA workshop. SANDA stands for Supplying Accurate Nuclear Data for energy and non-energy Applications. Accurate nuclear data are of fundamental importance in a number of different nuclear and non-nuclear fields. The EU-funded SANDA project aims to produce a data library containing high-quality nuclear measurements on key isotopes and reactions. The project puts special focus on how data is measured, evaluated and validated. These high-accuracy nuclear data can be exploited by safety authorities, research institutions, the nuclear industry and health organisations. SANDA aims to widely and efficiently disseminate its results through the Nuclear Energy Agency (NEA) and the International Atomic Energy Agency (IAEA).

Within SANDA, I am involved in the coordination of preparation of nuclear targets for high-quality nuclear measurements on key isotopes and reactions. The actinide targets are samples consisting of a freestanding or a supported actinide material on a substrate and are used for nuclear reaction studies. Beside the high quality of these actinide targets, an accurate characterization is mandatory to know the number of atoms per unit area of the isotope of interest, the homogeneity and the presence of contaminants and impurities influencing the determination of reaction cross-section measurements.

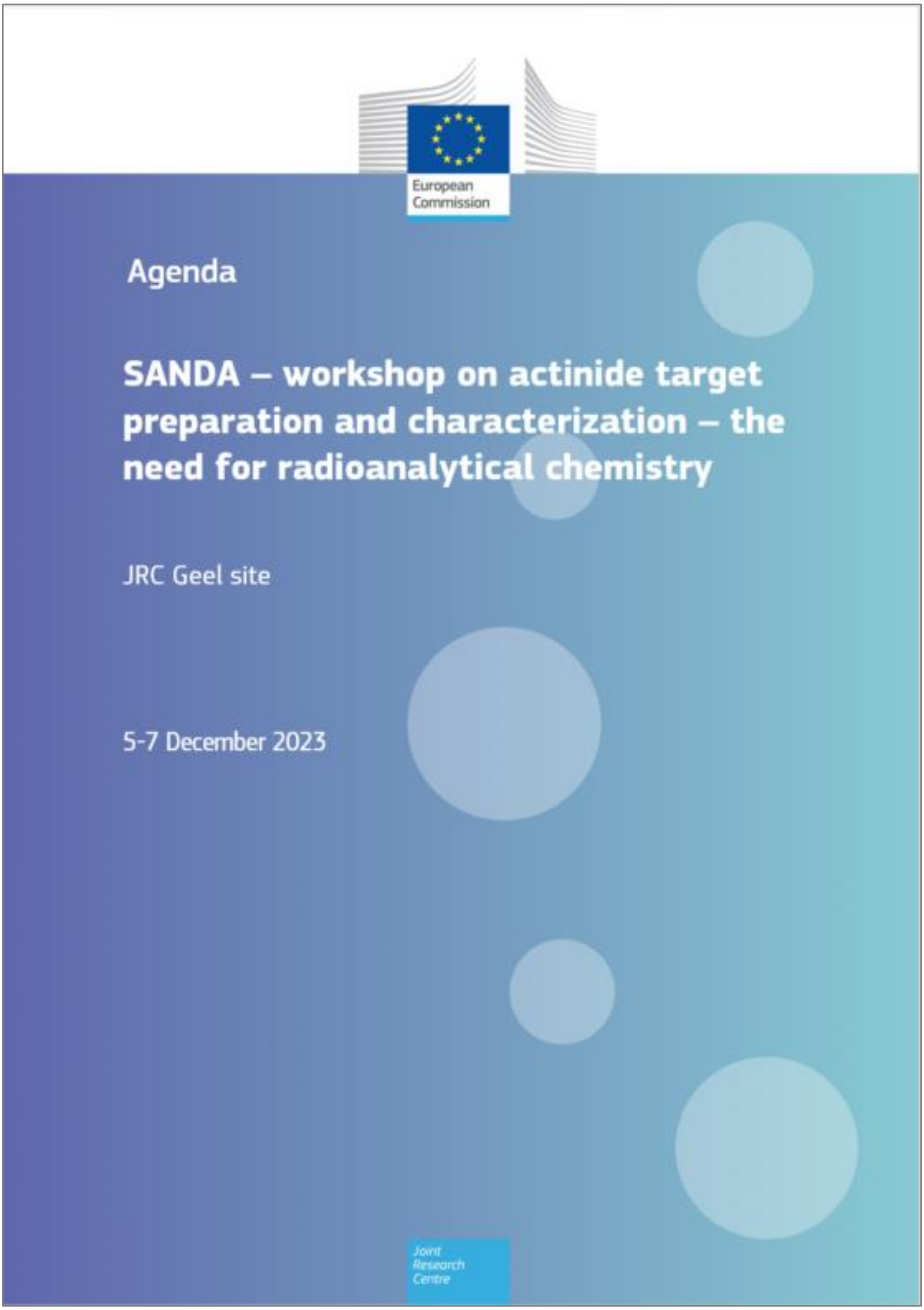
The demand for tailor made high-quality and well-characterized actinide targets for nuclear data measurements is high. Only a small group of laboratories in Europe has the knowledge and capacity to work with actinide material.

The aim of this workshop is to share knowledge among the actinide sample production and radioanalytical chemistry laboratories. The use of this actinide material including good characterization by radioanalytical chemistry has various applications across different domains: Nuclear Fuel, Nuclear Research, Nuclear Medicine, Environmental Monitoring, Nuclear Waste Management, Space Exploration, Security and Safeguards etc. Looking for synergies and creating space for networking will help us to become more efficient in high-cost and manpower intensive work.

The workshop is in person (not online) in order to have good detailed discussions and interactions.

I would like to thank the European Union for the financial support.

Goedele Sibbens



Agenda

SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

JRC Geel site

5-7 December 2023

SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

1st day: 5 December 2023

08:30-09:00	Arrival at the Joint Research Centre / Registration
09:00-09:30	Nuclear Data and Measurement Standards **Arjan Plompen Unit Head of G.II.5, JRC Geel, Belgium**
09:30-10:00	The SANDA project: high quality targets for successful experiments **Daniel Cano-Ott, CIEMAT, Spain**
10:00-10:30	Nuclear fuel research in the Belgian disposal context **Marc Verwerft, SCK CEN, Belgium**
10:30-11:00	Coffee break
11:00-11:30	JRC Geel target preparation laboratory **Goedele Sibbens, JRC Geel, Belgium**
11:30-12:00	Actinide target production methods for applications in chemistry and physics **Ernst Artes, JGU Mainz**
12:00-12:10	Group photo – Reception of Bldg 200
12:10-14:00	Lunch in cafeteria
14:00-14:30	Separation of Ra-Ac-Th and preparation of Ra targets **Stephan Heinitz, SCK CEN, Belgium**
14:30-15:00	Chemical purification and preparation of deposits by molecular plating **Jolanta Karpinska, JRC Geel, Belgium**
15:30-16:00	Coffee break
16:00-16:30	Celebrating 60 years of synthesis excellence, JRC Karlsruhe's contributions to transuranium elements and innovative material synthesis **Rachel Eloirdi, JRC Karlsruhe, Germany**
16:30-17:00	Revisiting the MALIBU project on high burnup UO_2 and MOX fuels **Marc Verwerft, SCK CEN, Belgium**

2nd day: 6 December 2023

08:30-09:00	Arrival at the Joint Research Centre
09:00-09:30	Analytical methods for actinide material analyses at the JRC Karlsruhe **Razvan Buda, JRC Karlsruhe, Germany**
09:30-10:00	Radiochemistry and mass spectrometry **Ben Russell, NPL, UK**
10:00-10:30	Radiochemical analysis application and challenges for actinides **Ingrid Geuens, SCK CEN, Belgium**
10:30-11:00	Coffee break
11:00-11:30	Accelerator mass spectrometry of actinides – radiochemical separations and capabilities **Sebastian Fichter, HZDR, Germany**
11:30-12:00	Nuclear chemistry at Chalmers University of Technology – capabilities and research areas **Marcus Hedberg, Chalmers University of Technology, Sweden**
12:00-14:00	Lunch in cafeteria
14:00-14:30	Challenges of radiochemical separation methods for actinides **Mirela Vasile, SCK CEN, Belgium**
14:30-15:00	Update on the application of extraction chromatographic resins in the separation of actinides **Steffen Happel, Triskem International, France**
15:30-16:00	Coffee break
16:00-16:30	Nuclear chemistry and radioanalytics at CTU in Prague **Mojmír Němec, Czech Technical University, Czech Republic**
16:30-17:00	Characterization of actinide targets at JRC- Geel **David Vanleeuw, JRC Geel Belgium**
18:00 -21:30	Dinner **Tabloo, Dessel**

3rd day: 7 December 2023

08:30-09:00	Arrival at the Joint Research Centre
09:00-09:30	Characterization of actinide targets **Klaus Eberhardt, JGU Mainz, Germany**
09:30-10:00	Electron microscopy studies on nuclear material **Antonio Bulgheroni, JRC Karlsruhe, Germany**
10:00-10:30	Coffee break
10:30-11:00	Target laboratory @ GSI and FAIR **Bettina Lommel, GSI Darmstadt, Germany**
11:00-11:30	The need for an actinide laboratory **Christelle Stodel, GANIL, France**
11:30-12:00	Closing
12:15-13:30	Lunch in cafeteria
13:45-16:00	Visit of Laboratories
16:40	Departure

SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

Photo with the workshop participants

SANDA

SANDA

Supplying Accurate Nuclear Data for energy and non-energy Applications

HORIZON2020

From left to right and from the first row up to the fourth row:

- J. Karpinska, G. Sibbens, D. Vanleeuw, A. Plompen, Y. Aregbe, R. Eloirdi, M. Verwerft
- D. Cano Ott, K. Eberhardt, S. Fichter, M. Němec, S. Heinitz, S. Oberstedt, A. Ernst, B. Kindler
- L. Viera, M. Hedberg, S. Happel, B. Lommel, M. Vasile, C. Stodel, C. Paradela, P. Schillebeeckx, A. Moens, B. Ballester
- R. Buda, A. Bulgheroni

Not in the photo: R. Taylor, I. Geuens

SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

Abstracts

presented on 1st day: 5 December 2023

THE SANDA PROJECT: HIGH QUALITY TARGETS FOR SUCCESSFUL EXPERIMENTS

D. Cano-Ott

CIEMAT

Accurate nuclear data are of fundamental importance in numerous nuclear and non-nuclear fields. The EU-funded SANDA project aims to create a data library comprising high-quality nuclear measurements on key isotopes and reactions. This high-precision nuclear data can be utilized by safety authorities, research institutions, the nuclear industry, and health organizations. SANDA intends to disseminate its results widely and efficiently through the Nuclear Energy Agency (NEA) and the International Atomic Energy Agency (IAEA).

The SANDA project places special focus on the measurement of data. To achieve this, SANDA supports the realization of new measurements using improved detectors, techniques, and high-quality targets. The quality of a sample or target to be irradiated is of paramount importance in any nuclear data experiment, as it directly influences the accuracy and reliability of the results. Samples are tailored for each type of experiment, and their production typically requires well-equipped radiochemistry laboratories, well-trained scientific and technical personnel, and sophisticated instrumentation. The material for experiments needs to be provided in sufficient amounts, free from interfering impurities, well-characterized, and in a suitable physical form. Given that most of the desired samples and targets consist of exotic and rare materials, their production is costly and requires significant manpower.

A general overview of the SANDA project and practical examples of experiments involving samples produced within it will be provided during the workshop.

NUCLEAR FUEL RESEARCH IN THE BELGIAN DISPOSAL CONTEXT

Marc Verwerft

Belgian Nuclear Research Centre (SCK CEN), Boeretang 200, B-2400, Mol, Belgium

Earlier this year, the Belgian government and the nuclear operator of Belgian's seven nuclear power plants (ENGIE) reached an agreement in principle on the lifetime extension of the two most recent nuclear power plants. Doel 4 and Tihange 3 that were scheduled to shut down in 2025 are now proposed for a lifetime extension of ten years. The final agreement is expected to be soon concluded. The agreement in principle involves also agreements on the transfer of liabilities for the nuclear waste and spent fuel from the operator to the Belgian State. Spent nuclear fuel comprises only a small fraction of the total volume of nuclear waste to be disposed of, but contains the vast majority of the total radioactivity and in all aspects presents the highest technological challenges. It requires shielding in all handling stages, continues to generate decay heat that needs to be dissipated, must be configured in a manner that excludes criticality under any circumstance, etc. Although the implementation of final disposal of the spent nuclear fuel is scheduled decades from now, the imminent transfer of these liabilities incited a significant acceleration of efforts in the domain. Until now, NIRAS/ONDRAF was responsible for disposal concepts and related R&D work, while ENGIE controlled the inventory and was responsible for the nuclear fuel in its operational context. Since working towards the final disposal becomes now concrete, these two axes need to merge.

Final disposal in poorly indurated ("soft"), geologically stable clay layers is Belgian's reference scenario for high level vitrified waste and irradiated nuclear fuel when declared as waste. Both waste types would be loaded in multi-barrier containers prior to disposing them in underground, stable, clay layers. Spent fuel degradation, leaching of radionuclides from the spent fuel, sorption and precipitation are the innermost near-field effects that start when the containment period of the engineered barrier comes to its end. Research in this field has started in 1974 with the intiative of SCK CEN to launch a program on geological disposal, and since the early 1980's, soon after the creation of the underground facility HADES, this R&D program was gradually taken over by NIRAS/ONDRAF, and is today entirely managed by NIRAS/ONDRAF. This research will continue to further refine our understanding, develop and validate models to support the decision making process for the final disposal, its licensing and technical implementation.

With the exception of spent fuel from SCK CEN's test reactor (BR2), Belgium suspended reprocessing of spent nuclear fuel since December 1992. The suspension of reprocessing of SNF from power reactors has, however, not lead to its declaration as waste, hence remained in the operator's liability and under its operational command. After the initial cooling period in the spent fuel docks of the NPPs, spent fuel is transferred to dry storage vaults at Doel or a centralized wet storage facility in Tihange.

Spent nuclear fuel is characterized by detailed, static data characterizing each assembly with today's known information. These data are derived from the fuel management system via code calculations that use fuel assembly fabrication, core management, storage and handling data as input. The fuel management system which is today used in the context of operation, must in the future be used from a disposal perspective. In the decade to come, Belgium will be confronted first with the transfer of data and knowledge from the nuclear operator (ENGIE) to the nuclear waste management agency (NIRAS/ONDRAF) and later with the challenge to enrich these data with information that was not necessarily relevant in an operating context, but will be from a disposal perspective.

Experimental research is conducted to advance understanding, develop models and validate calculation results. The principal input parameters (radiological source term, decay heat and margin to criticality) are obtained from inventory calculations via identical physics codes that are used for reactor core calculations. Other parameters (fission gas release, cladding corrosion, physical state of the nuclear fuel) are obtained from fuel performance codes that use the irradiation history and fuel fabrication data as main input. Long term fuel degradation upon exposure to specific environmental conditions (pH, dissolved hydrogen content) is estimated using dedicated thermodynamic-kinetic codes.

Properties, timescales and key nuclides may differ between an operating and a disposal context, but both in an operating and in a disposal context, nuclear fuel management heavily relies on calculations that involve nuclear data, methods and codes. Continued efforts to improve methods and codes, quantify and where possible reduce uncertainties, and validate results remain crucial to the cautious management of spent nuclear fuel for the century to come.

PREPARATION AND CHARACTERIZATION OF ACTINIDE TARGETS AT JRC-GEEL

G. Sibbens, D. Lewis A. Moens, G. Sibbens, D. Vanleeuw
European Commission, Joint Research Centre (JRC), Geel, Belgium
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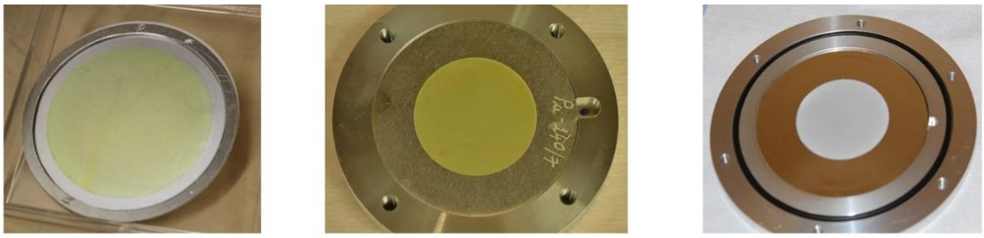
The JRC-Geel target preparation laboratory of the Joint Research Centre prepares and characterizes a range of tailor made targets for neutron-induced reaction measurements, e.g. cross-sections and particle emission yields. An overview of the different techniques with examples is described in [1].

For the preparation of thin layer actinide targets, two techniques are applied: molecular plating and physical vapour deposition. Molecular plating is a cathodic deposition of the actinide material (U-, Pu-, Am-, Th- and Np-isotopes) in an isopropanol solution onto an aluminium substrate (Fig. 1). For each isotope, a cell is fabricated in the JRC-Geel workshop according to the required dimensions of the substrate and the deposit. The second technique, physical vapour deposition is a process whereby the source material is heated in a vacuum in order to increase the vapour pressure, allowing the vapour to expand and condense on a colder substrate of plastic or metal. In the case of thermal evaporation of $^{235}UF_4$ and $^{238}UF_4$ (Fig. 2), resistance heating is used and for $^{235}U_3O_8$, electron-beam evaporation is applied. In case the ingrowth of a daughter nuclide would interfere with the characterization and cross-section measurements, a radiochemical separation is done prior to the preparation of the layer. The areal density of the deposited material is calculated from the mass and the diameter of the deposit. The mass of the isotope of interest is derived from the alpha-activity measured by low-solid angle alpha-particle counting and the isotopic composition determined by the JRC-Geel mass spectrometry laboratory.

In addition, the laboratory also has the capability to prepare thick actinide samples like discs by punching and pellets by uniaxial pressing of powders. Polyimide foils with a typical areal density of 35 µg/cm^2 are produced by spin coating glass plates and in-situ poly-condensation and are used as substrate for some of the actinide thin layer targets. All targets are prepared in a nuclear controlled area in a glove box dedicated to the material and the oxidizing material in an argon glove box as far as possible.

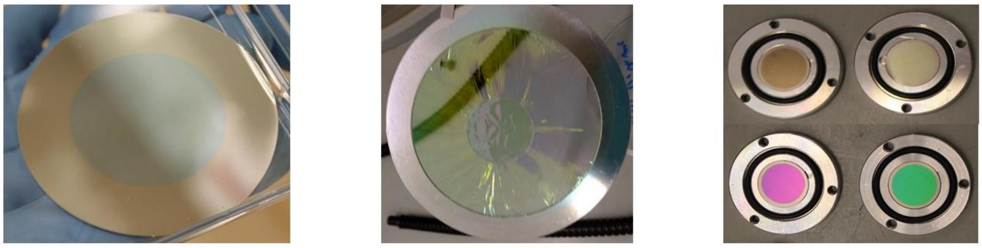


99.934% ^{235}U layer, Ø 42 mm, 363 µg·cm^{-2} deposited by molecular plating on 30 µm Al stretched on Al ring

99.9% ^{240}Pu layer, Ø 30 mm, 244 µg·cm^{-2} deposited by molecular plating on 0.25 mm Al disc

99.98% ^{241}Am layer, Ø 60 mm, 4.7 µg·cm^{-2} deposited by molecular plating on 25 µm Al stretched on Al ring

Fig. 1: Thin layers prepared by molecular plating



99.998% ^{238}U layer, 48 µg·cm^{-2} deposited on stainless steel

99.998% ^{238}U layer, 377 µg·cm^{-2} deposited on a polyimide foil

99.998% ^{238}U layer, from 12 up to 358 µg·cm^{-2} deposited on titanium substrate

Fig. 2: Thin layers prepared by physical vapour deposition

ACTINIDE TARGET PRODUCTION METHODS FOR APPLICATIONS IN CHEMISTRY AND PHYSICS

E. Artes[1,2,3], Ch. E. Düllmann[1,2,3], A. T. Loria Basto[1,2], C.-C. Meyer[1,2], C. Mokry[1,2], L. E. Reed[1,2], D. Renisch[1,2], J. Runke[1,3], N. Trautmann[1], V. Urbanova[1,2]

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Radioisotopes and especially the actinides have found wide application in scientific research projects. They serve as recoil ion sources [1-4], for various experiments to determine atomic and nuclear properties by laser spectroscopy (e.g., [5]) or by muon spectroscopy [6], as targets for nuclear data studies [7] or for the production of superheavy elements (SHE) at particle accelerators [8,9].

For each experiment, the requirements for the samples are different as far as geometry, thickness, chemical and physical properties, and the influence of a matrix are concerned. Thus, the samples and consequently their production methods must be tailored to the experiments, and there is no universal solution for all these issues. Therefore, different methods for producing these samples have been developed. These methods include dipping onto surfaces, electrochemical depositions such as molecular plating (MP) [1,2,10,11], or drop on demand (DoD) printing [4,12].

MP is a 60-years old technique, which was introduced by Parker and Falk 1962 [9] and is well established today. In MP, radioisotopes dissolved in minute amounts of dilute acid are mixed with organic solvents and then deposited using a constant current and high voltage of several hundred volts. Thin films of up to about 800 µg/cm^2 can be produced in a single deposition step [1,2,10]. These films are macroscopically homogeneous but show cracks and tiles under the microscope. The thin films produced in this way show strong adhesions to the surface and are used, e.g, as targets in the production of SHEs. Although the method has been known for so long, it is still not fully understood [13], neither concerning the exact mechanism nor the chemical composition of the produced thin films [1,2,10]. The method of electrochemical deposition must be improved, both regarding film thickness as well as beam resistance [1], to cope with increased ion beam intensities and to achieve higher production yields. Therefore, various spectroscopic methods were recently applied to MP targets before and after ion beam irradiation, including Raman spectroscopy [14], XPS [15], and various ion beam analysis methods. The transfer to more modern electrochemical methods developed for f-elements appears promising as well [16].

Complementary to MP, DoD printing is a method frequently used in our lab. It relies on the production of nanoliter-sized drops that fall onto a surface where they quickly evaporate [12,13]. Advantages of DoD include the possibility to deposit thin films on non-conductive surfaces and on very thin and delicate substrates that do not tolerate the mechanical stress associated with the MP method. The method is extremely flexible, as it is possible to print both aqueous and organic solutions. The salts of the radioisotopes can be varied and additives such as polymers can be added for further treatment of the thin films after deposition, such as thermal combustion. This also allows relatively homogeneous thin films to be produced. There is also much freedom concerning specific shapes or geometries that the deposits need to have (as an example, see Fig. 2b in [17]).

At the workshop, an overview of target production and analytics performed in our group will be presented.

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[2] K. Eberhardt et al. AIP Conf. Proc. 1962, 030009 (2018)
[3] L. von der Wense et al. Nature 533, 47-51 (2016)
[4] R. Haas et al. Radiochim. Acta 108, 923-941 (2018)
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[12] D. Renisch et al. EPJ Web of Conferences 285, 04001 (2023)
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[14] C.-C. Meyer et al. Nucl. Instrum. Methods. Phys. Res. A, 1028, 166365 (2022)

SEPARATION OF RA-AC-TH AND PREPARATION OF RA TARGETS

S. Heinitz[1], B.Ballister[1,3], A. Cea[1], D. Maertens[1], J. Mermans[1] J. Wagemans,[1] A. Stankovsky[1], A. Tsinganis[2], J. Heyse[2], A. Plompen[2], M. Kerveno[3]

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[15] A. Vascon et al. Nucl. Instrum. Methods. Phys. Res. A, 696, 180-191 (2012)
[16] C.-C. Meyer et al. Radiochim. Acta (2023) https://doi.org/10.1515/ract-2023-0197
[17] S. Rau et al., Nature 585, 43-47 (2020)

The isotope ^{225}Ac has gained significant attention in nuclear medicine in the recent years as possible targeted alpha therapy nuclide for the treatment of different cancer types. Production of this isotope can be achieved through a variety of pathways [1], of which the milking from a ^{229}Th generator is the most convenient. Given the extremely scarce availability of ^{229}Th worldwide, SCK CEN has engaged in the recovery of that isotope from existing legacy material existing on site [2,3]. Substantial quantities of ^{227}Ac and ^{226}Ra also present in this material required precautions during handling due to the emanation of radon (^{219}Rn and ^{222}Rn) and the considerable dose rate. This contribution aims to share the experience in processing this legacy material in a glove-box containing large quantities of alpha emitters.

In order to establish an independent ^{225}Ac production pathway, efforts are undertaken in parallel towards photonuclear transmutation of ^{226}Ra to enable a larger availability of ^{225}Ac in the future. The aim is the indirect production route via the ^{226}Ra(γ,n)^{225}Ra(β-)^{225}Ac reaction using Bremsstrahlung photons from an electron accelerator. Due to the lack of relevant nuclear cross section data for the ^{226}Ra(γ,n)^{225}Ra reaction, a collaboration has been established in collaboration with JRC Geel, Belgium, for dedicated irradiations of ^{226}Ra using the GELINA electron accelerator. For this purpose, thin, uniform and radon tight targets need to be prepared using the molecular plating technique, which is currently tested with Ba as homologue for the development of the target preparation method. First target preparation results will be shown that were obtained with stable barium, serving as basis for future irradiation experiments involving ^{226}Ra in order to enable measurements on the ^{226}Ra(γ,n)^{225}Ra cross section.

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[2] L.H. Baetslé, et al.: Gram scale production of 227Ac from irradiated radium; Proceedings of the second international symposium on power from radioisotopes; Madrid, pp. 95 – 119 (1972).
[3] S. Boden et al.: Thorium-229 quantified in historical Thorium-228 capsules; *Applied Radiation and* Isotopes 120, pp. 40 – 44 (2017).

CHEMICAL PURIFICATION AND PREPARATION OF ACTINIDE DEPOSITS BY MOLECULAR PLATING

J. Karpinska, D. Lewis A. Moens, G. Sibbens, D. Vanleeuw
European Commission, Joint Research Centre (JRC), Geel, Belgium
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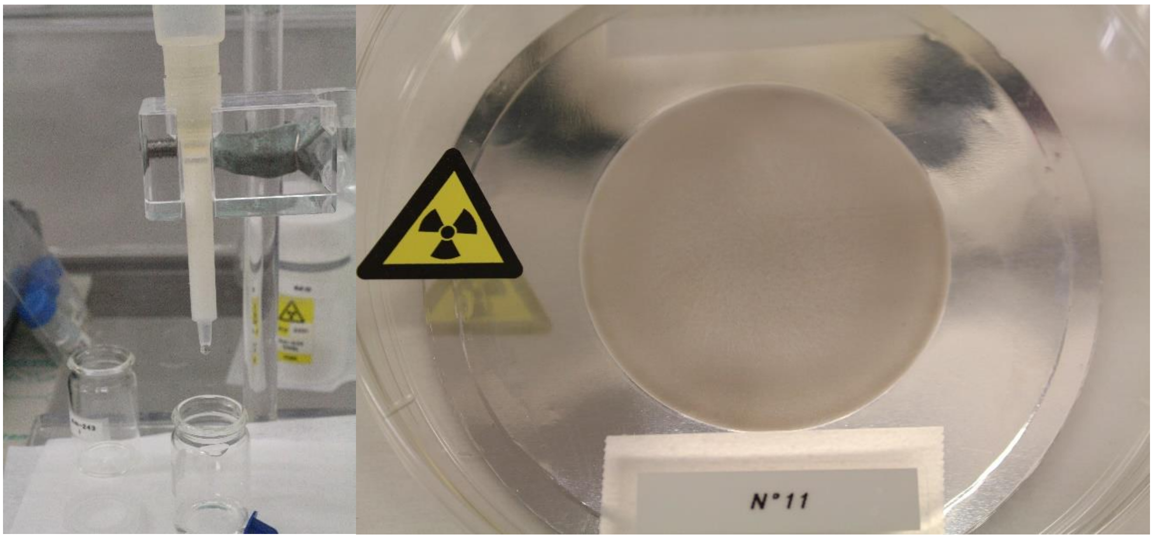
The mission of the Target Laboratory at JRC-Geel is the production of actinide targets for nuclear data measurements. The presence of impurities in the target of interest, such as the ingrowth of daughter products, often hampers the study of nuclear reactions[1]. Therefore, chemical purification is frequently required to produce high-quality targets that meet user-defined specifications. This presentation will focus on the most recent requests for the production of ^{243}Am and ^{241}Pu targets. For fission cross-section measurements, thin and thick deposits of ^{243}Am with the areal density of 3 µg/cm^2 and 150 µg/cm^2 were prepared via molecular plating, Fig 1. ^{243}Am was purified via extraction chromatography using DGA (N,N,N′,N′-tetra-n-octyldiglycolamide) resin. DGA resin was reported to have high affinity for Am and were successfully employed in the separation of Am from Pu and other actinides[2], [3]. Further, the current status of ongoing target requests: the chemical purification and preparation of thin layers of ^{241}Pu for the measurement of neutron capture and fission reactions, will be discussed.

Fig. 1: Purification of ^{243}Am via column extraction chromatography and ready ^{243}Am target.



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[1] H. Mast, R. Eykens, J. Pauwels, C. Wagemans, *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment* **1989**, *282*, 107-109.

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CELEBRATING 60 YEARS OF SYNTHESIS EXCELLENCE, JRC KARLSRUHE'S CONTRIBUTIONS TO TRANSURANIUM ELEMENTS AND INNOVATIVE MATERIAL SYNTHESIS

Rachel Eloirdi, Olaf Walter, Karin Popa, Pavel Soucek

European Commission Joint Research Centre, P.O. Box 2340, 76125 Karlsruhe, Germany

In this presentation we will highlight the significant achievements of JRC Karlsruhe over its 60-year history, focusing on its expertise in handling transuranium elements and its contributions to the development of various synthesis methods. The institute has established a strong reputation in the field, particularly in the synthesis of actinide-based compounds, nanoparticles [1], organometallics [2], and the synthesis of single crystals from intermetallic to oxides.

The diverse range of synthesis techniques employed at JRC Karlsruhe will be discussed, including traditional methods and innovative approaches such as 3D printing for the production of target materials used in medicine radionuclide production. These synthesis methods have played a crucial role in advancing the field and have opened up new possibilities for material synthesis.

In addition to synthesis, the characterization of samples is essential to define their microstructure and properties in physics or thermodynamics. JRC Karlsruhe recognizes the importance of thorough characterization and emphasizes its significance in understanding the synthesized materials but also to the role plaid by the radiochemical analysis in this context.

An overview of JRC Karlsruhe's contributions to the field of synthesis and characterization will be presented. It showcases the present institute's expertise in handling transuranium elements and highlights its achievements in the development of various synthesis methods. By exploring these advancements, researchers can gain valuable insights and inspiration for their own work in the field of material synthesis.

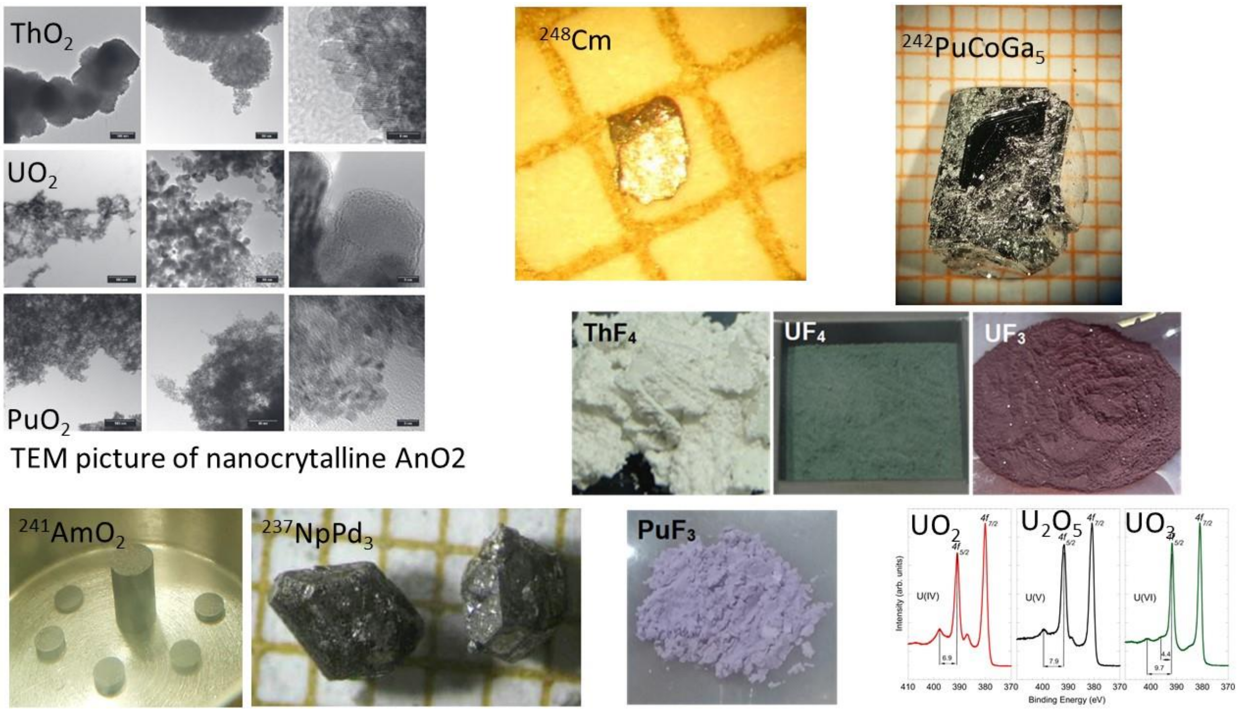

Fig. 1: Examples of samples produced at JRC Karlsruhe, from oxide to intermetallique, in powder, pellet or single crystals forms and thin films X-ray photoelectron spectroscopy analyses.

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SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

Abstracts

presented on 2nd day: 6 December 2023

ANALYTICAL METHODS FOR ACTINIDE MATERIAL ANALYSES AT THE JOINT RESEARCH CENTRE KARLSRUHE

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Safeguarding of nuclear material strongly depends on state of the art analytical performance. It is crucial for inspectors to receive timely analytical results with lowest achievable uncertainty for facilities handling bulk material. The Directorate-General Energy (ENER) fulfils the safeguards obligations stipulated by the Article 77 of EURATOM treaty for European facilities. The Joint Research Centre (JRC) carries out destructive and non-destructive analyses of nuclear material on the site of JRC-Karlsruhe, on the site of the reprocessing plant from Orano, at La Hague, France as well as in campaigns at fuel fabrication facilities in Europe.

The JRC is operating analytical instruments fit for purpose, ensuring continuity and quality control of the generated analytical results. The quality of these results is monitored through participating in inter-laboratory comparison exercises and by the use of traceable reference materials. The results obtained in the exercises substantiate the position of the JRC-Karlsruhe between the top performing laboratories world wide for analyses of actinides like uranium, plutonium, americium but also impurities in various actinide matrices.

The Analytical Srvice (AS) of the JRC-Karlsruhe is ISO 17025 accredited and provides a vast array of methods for analyses of nuclear samples under the scope of the accreditation and further analyses beyond the scope of accreditation.

The main methods are shown in Tabble 1.

RADIOMETRIC METHODS		DESTRUCTIVE ANALYSIS	
High Resolution Gamma Spectrometry (HRGS)	Non-destructive analysis of gamma emitting nuclides	Thermal Ionisation Mass Spectrometry (TIMS) and Isotope Dilution-TIMS	Measurement of isotopic composition of U and Pu and high precision determination of U and Pu content (ID-TIMS)
Hybrid K-edge / X-Ray Fluorescence Densitometry (HKED)	Determination of U and Pu content in liquid samples - JRC-Karlsruhe development	Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	Determination of concentrations and isotopic abundances of (almost all) chemical elements at very low levels
COMbined Procedure for Uranium Concentration and Enrichment Assay (COMPUCEA)	Mobile U elemental content and 235U enrichment determinations in fuel fabrication plants - JRC-Karlsruhe development	ELemental ANALysis of light elements (ELANA)	Elemental analysis of C, O and N
Calorimetry	Pu and Am mass determination	Large Geometry - Secondary Ion Mass Spectrometry (LG-SIMS)	Determination of U isotopic composition of uranium particles in the search for undeclared nuclear activities
Neutron Coincidence Counting (NCC)	non destructively assay the concentration of Pu in High Burn-up MOX pellets		

Fig. 1: The analytical methods available at the JRC-Karlsruhe

The JRC strives for continuous improvement through ambitious R&D projects in order to keep up with global evolving technologies and cutting edge scientific developments. The performance of the laboratory and the latest developments will be presented during the workshop.

RADIOCHEMICAL ANALYSIS
APPLICATION AND CHALLENGES FOR ACTINIDES

Dr. Ingrid Geuens, Dr. Nancy Wanna

SCK CEN

There are many different technologies available that are used in radiochemical analysis. Several mass spectrometry techniques (e.g. RQ-, TQ-, SF-ICP-MS, TIMS), radiometric spectrometry techniques such as alpha, gamma as well as optical emission and microstructural techniques (e.g. SEM, EBSD, EPMA) along with dedicated sample preparation are used. Several of these techniques are also used in standard analytical laboratories. However radiochemical analysis face several additional challenges, including safety precaution for working with high radioactive materials, special precautions during samples preparation and adaptations to the instruments to work in a safe environment. For example, performing radiochemical analysis on radioactive samples involves working with hot cells and glove boxes.

Fig. 1: HPIC-SF-ICP-MS in controlled area for on-line separation and concentration determination of actinides.

Quantitative determination of nuclides, particularly unstable nuclides, requires special analysis strategies taking into account background interferences in radiometric spectrometry techniques due to counting statistics. Specifically for mass spectrometry techniques, isobaric interferences of nuclides or polyatomic molecules at the mass of the nuclide of interest is a common notorious limitation [1]. Seeing that the materials of interest to radiochemical analysis are not of natural origin, correction with other masses is not possible. Sample preparation using dedicated separation can help to overcome these isobaric interferences. For example, on-line chromatographic separation, such as high performance ion chromatography HPIC, offers a fast and safe way of working [1 – 3]. In order to get uncertainties as low as possible, a combination of on-line chromatographic separation with isotope dilution mass spectrometry (IDMS) is used to obtain accurate and precise results which enables scientists to develop new radioisotopic standards, characterise fuels, spent nuclear fuels and waste.

An example will be presented for the quantitative determination of Am mass fraction and isotopic composition. Using isotope dilution HPIC-SF-ICP-MS, the isobaric overlap of Pu and Am nuclides (such as ^{241}Am and ^{241}Pu) is eliminated and the required level of uncertainty is achieved.

Another example is the determination of Pu isotopic composition (see Table 1 below).

Isotope	Mass fraction by HPIC-SF-ICP-MS (100)	U (k=2)
$m(^{238}\mathrm{Pu})/m(\mathrm{Pu})$	3.819	0.012
$m(^{239}\mathrm{Pu})/m(\mathrm{Pu})$	41.508	0.038
$m(^{240}\mathrm{Pu})/m(\mathrm{Pu})$	31.418	0.044
$m(^{241}\mathrm{Pu})/m(\mathrm{Pu})$	7.783	0.025
$m(^{242}\mathrm{Pu})/m(\mathrm{Pu})$	15.468	0.057
$m(^{244}\mathrm{Pu})/m(\mathrm{Pu})$	0.00264	0.00023

Table 1: Example of the determination of Pu isotopic composition using HPIC-SF-ICP-MS.

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ACCELERATOR MASS SPECTROMETRY OF ACTINIDES RADIOCHEMICAL SEPARATIONS AND CAPABILITIES

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Helmholtz-Zentrum Dresden-Rossendorf, Institute of Ion Beam Physics and Materials Research

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 (**H**elmholtz **A**ccelerator **M**ass **S**pectrometer **T**racing **E**nvironmental **R**adionuclides) 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.

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. Different radiochemical separation schemes for actinides (i.e. U, Pu) and other radionuclides are in routine operation[5] whilst the separation of other actinides (Pa, Np, Am/Cm) is currently under development. In this talk different radiochemical work-up procedures will be presented and the future capabilities of the HAMSTER facility are highlighted.

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NUCLEAR CHEMISTRY AT CHALMERS UNIVERSITY OF TECHNOLOGY – CAPABILITIES AND RESEARCH AREAS

Marcus Hedberg
Chalmers University of Technology

Nuclear research and scientific exploration differs from many other areas of research in one clear aspect. Due to the inherent nature and properties of radioactive materials generally and nuclear materials specifically, special considerations need to be considered in order to work with these materials. The result of this is that nuclear chemistry and other radiation sciences find it challenging to co-exist with 'general' chemistry from a pure laboratory aspect. The non-radioactive sciences typically don't desire radioactive materials in their laboratories. This has resulted in that many countries have opted for organizing radiation-based research through the means of dedicated national laboratories/facilities rather than through extensive infrastructure located at universities.

Sweden differs from many European countries in the sense that there is no dedicated national facility for conducting nuclear chemistry research. Research in such areas is rather carried out by the universities and the universities doing research in nuclear sciences need to have their own infrastructure dedicated and dimensioned for the radioactive material quantities they are working with. Currently there is only one university group in Sweden possessing a permit for operating a facility allowed and capable of doing research on transuranium actinides and that is the nuclear chemistry group at Chalmers University of Technology.

This presentation will show both the laboratory premises and capabilities of the Chalmers laboratories including radio-analytical competences together with highlighted areas of research that we have been and currently are interested in.

CHALLENGES OF RADIOCHEMICAL SEPARATION METHODS FOR ACTINIDES

Vasile M., Jacobs K., Bruggeman M.

SCK CEN, Low-Level Radioactivity Measurements Expert group

The complex chemistry of actinides makes their radiochemical separation challenging. Additionally to this, the selection of the measurement approach, such as alpha particle spectrometry or ICP-MS, adds other challenges related to the specific sample preparations for these techniques. Alpha particle spectrometry needs a pure fraction of the actinide to be measured and a very thin source for best spectral resolution that can be obtained by electrodeposition [2] or by micro-precipitation. Mass spectrometry may suffer from isobaric interferences which have to be removed during radiochemical separation.

In this presentation, advantages, disadvantages and associated challenges of a sequential separation approach for determination of ^{237}Np, ^{241}Am, thorium, plutonium, and uranium isotopes will be discussed [1]. This presentation is based on the manuscript of Vasile et al., 2017 [1].

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UPDATE ON THE APPLICATION OF EXTRACTION CHROMATOGRAPHIC RESINS IN THE SEPARATION OF ACTINIDES

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[2]RadAnal Ltd, Budapest (Hungary)

Due to their generally high selectivity and rather fast kinetics extraction chromatographic resins are very frequently finding use in the separation of actinides from a variety of samples/matrices. The availability of resins with varying selectivity's further makes the combination of two or more resins possible (e.g. as stacked cartridges) allowing the separation of several actinides from one sample. A short overview over the structures and general selectivity of the four extraction chromatographic resins mainly used in actinide separation chemistry (UTEVA, TEVA, TRU and DGA Resins) will be given. Focus will then be shifted towards two new extraction chromatographic resins (TK200 and TK221) that are increasingly finding use in this context.

TK200 Resin is an extraction chromatographic resin that is based on trioctylphosphine oxide (TOPO) which has some characteristics that make it suitable for use in actinide separation chemistry. In general, its selectivity is similar to the UTEVA resin as it retains uranium and tetravalent actinides, compared to UTEVA it shows significantly higher U and Th uptake though [1]. This, and the fact that Pu(III), like Am(III), is not retained from acidic media results, amongst others, in an increased use of the TK200 Resin for U removal from samples before Pu quantification by mass spectrometry [2], including work on the determination of 238Pu, 239Pu, 240Pu and 241Pu by ICP-MS/MS [3]. In the latter case a U decontamination of > $2\cdot10^9$ could be obtained using two TK200 cartridges.

A somewhat unique feature of the TK200 Resin is that it also shows very high retention of uranium, thorium and plutonium (IV) at very low acid concentrations (e.g. pH 2) making it suitable for their direct preconcentration from acidified water samples. It could be shown by Nora Vajda et al [1] that it is possible to concentrate U, Pu and Th from ~1L of acidified water samples on a 2 mL cartridge of TK200. The optimization of a sequential separation of these actinides on TK200 is currently on-going. Youyi Ni et al. applied a similar strategy to the determination of Pu in water samples [4] including the automation of the separation.

TK221 Resin is comprised of a mixture of the TO-DGA extractant and a phosphine oxide [5]. While it currently is mainly used in Ac-225 separation from irradiated Ra-226 targets or generator eluates [6] it also shows some interesting properties with respect to its selectivity for actinides, as could be shown by Papp et al. [7]. Mainly, the TK221 has higher retention of U than the DGA Resin and higher Am retention than TRU Resin. Papp et al. showed that TK221 can be used, in combination with TEVA Resin, to separate U/Am and Pu/Th from water samples, including sea water, with high recoveries.

Lastly, a short update on the development of a new impregnated membrane for the retention and measurement of actinides from water samples will be given.

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NUCLEAR CHEMISTRY AND RADIOANALYTICS AT CTU IN PRAGUE

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Department of Nuclear Chemistry (DNC) of the Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague is a university department with broad research activities ranging from radioecology through radiation chemistry, chemistry of the nuclear fuel cycle, up to ultratrace analysis and superheavy elements research. Regarding the topic of the SANDA workshop, DNC is equipped with standard set of radiometric devices for liquid scintillation counting, alpha spectrometry, general and low-energy gamma ray spectrometry, all of them available in the controlled area in the class B radiochemical laboratories. For higher activities of shorter-lived radionuclides two semi-hot cells are available, glove boxes for alpha nuclides are planned.

DNC participated in the series of P&T projects (EUROPART, GENIORS, ACSEPT, SACSESS, FREDMANS...) being part of the development and testing of separation agents and systems for actinides partitioning [1]. In parallel, DNC is a member of a project aiming on production of radiopharmaceutical radionuclides including their fast, high purity separation [2]. In the radioanalytics, DNC is active in two main directions – development of various materials for radiochemical separations including their limited production (collaboration with Triskem company) [3]; and related both inorganic and organic syntheses, and ultratrace analyses using accelerator mass spectrometry including development of separation methods and target sample preparation for AMS [4]. In the radiation chemistry, DNC is producing various pure nanomaterials with sorption and mainly scintillation properties and tailored composition via radiation (UV, gamma, accelearated electrons) processes [5]. Through collaboration with Nuclear Physics Institute and UJV Rez, DNC has also access to AMS, SIMS, neutron activation analysis, and cyclotron beams (p, d, ^{3}He, ^{4}He) up to 50 MeV. The latest device is also used for projects aiming on chemical behaviour studies of homologues of superheavy elements, this is done in collaboration of DNC with Nuclear Physics Institute (Czech Republic), University in Oslo (Norway), Texas A&M University (USA), hosting also GSI Darmstadt [6], DNC part of this research aims on chemical studies in the liquid phase, developing fast microfluidic separations [7].

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ACTINIDE SEPARATIONS CAPABILITIES AT THE UK'S NATIONAL NUCLEAR LABORATORY

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The United Kingdom's National Nuclear Laboratory (NNL) has a wide range of capabilities related to actinide separations. The skills and capabilities we have in NNL can be traced back to the development of and support to industrial scale reprocessing and fuel manufacturing plants in the UK, such as Thorp at Sellafield. However, over the last ~15 years the focus has mainly been on research and development of advanced separation processes for future nuclear fuel cycles which recycle actinides in a closed cycle.

An exemplar project was the recent Advanced Fuel Cycle Programme (AFCP) which ran from 2019 to 2022; this was funded by the Department of Business Energy and Industrial Strategy (BEIS)[1] and led by NNL. As part of AFCP, a number of projects in the field of actinide separations were carried out. These included the development of new processes for spent fuel recycling such as Advanced PUREX (for separation of U, Pu and Np); innovative-SANEX (for separation of Am and Cm) and GANEX (for separation of U and transuranic actinides). The R&D programme covered all major aspects of the advanced reprocessing and minor actinide partitioning from dissolution to separations to conversion to fuel precursors, as well as the management of the secondary wastes generated from reprocessing. Whilst the programme mainly focused on advanced aqueous routes, it also enabled the rebuilding of skills and capabilities in molten salt based separations[2]. AFCP was highly collaborative. Whilst NNL focused mainly on development and testing of flowsheets in our PuMA (Plutonium and Minor Actinides) Laboratory, it also helped build capabilities at universities for more fundamental studies of actinide separations chemistry and technology development. Two examples are the establishment of multi-scale centrifugal contactor rigs at the University of Leeds for hydrodynamic testing and the development at the University of Lancaster of an innovative photochemical route for actinide conditioning which was ultimately tested with U and Pu at NNL's PuMA Lab.

NNL has also been an active and long-standing participant in European framework programme projects based on separation of actinides from spent fuels and wastes, from PYROREP and EUROPART in the 2000s through to PATRICIA and PUMMA projects now. Again, the focus has primarily been on development and testing of aqueous separation flowsheets.

At the Preston Laboratory, NNL has unique pilot-scale capabilities for processing uranic residues from the nuclear fuel cycle. Since 1995, more than 3000 drums of legacy materials have been processed through the laboratory, typically by solvent extraction in mixer-settlers. One example was the EAGL-RAPTOR process which used a tributyl phosphate (TBP) solvent extraction system coupled with additional complexant chemistry to ensure a very high degree of separation of uranium from gadolinium, neptunium, plutonium and technetium, allowing recycle of valuable material into fuel.

Over the last few decade in particular, NNL has extended its actinide separations capabilities from the nuclear fuel cycle to diverse applications including the recovery of americium-241 from aged plutonium dioxide for space power sources and isotopes such as lead-212 from historic uranium stocks for medical applications. New facilities to upscale these activities are presently in the design phase.

This presentation will provide a high level overview of these capabilities for actinide separations R&D at NNL.

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CHARACTERIZATION OF ACTINIDE TARGETS AT JRC-GEEL

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The mission of the Target Laboratory at JRC-Geel is the production and characterization of actinide targets for nuclear data measurements. Besides the quality of the prepared targets, accurate characterization for quantities, such as the total number of nuclei per unit area, effective area and homogeneity is essential in view of the qualitative output of these measurements and can have a strong impact on the total uncertainty of the quantities deduced from the measured data[1].
In this presentation, we will explain the characterization methodologies and techniques applied in the Target Laboratory in order to deliver the requested output. The methodology for the accurately definition of the areal density of the actinide of interest will be presented with a focus on low geometry alpha-particle counting as one of the applied techniques beside thermal ionization mass spectrometry. For the activity distribution, we explain and exemplify alpha scanning as well as gamma scanning, a technique recently introduced in the Target Laboratory. We also discuss the necessity to characterize for impurities and layer morphology and the techniques suitable for this purpose.

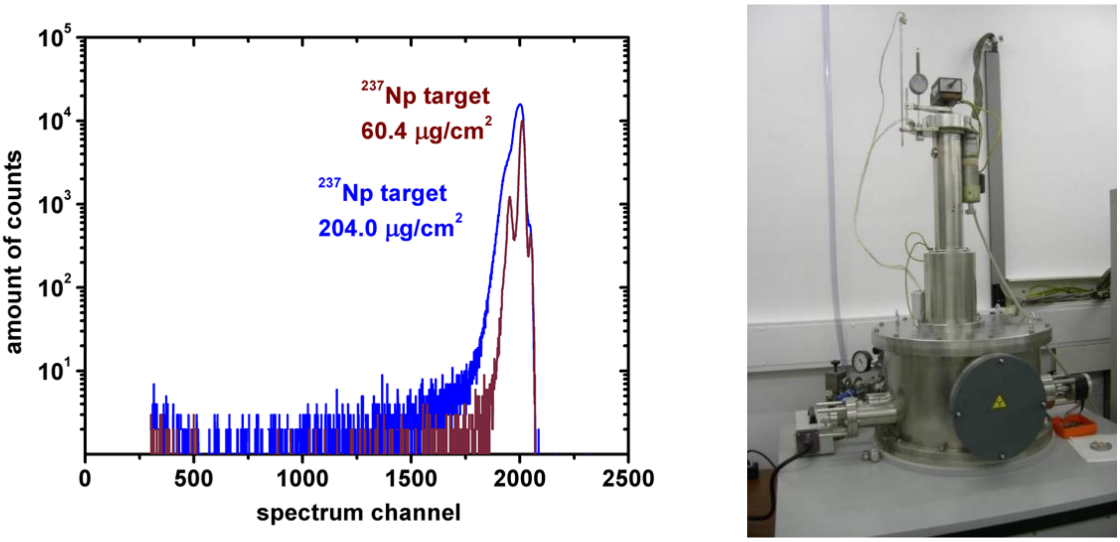

Fig. 1: Alpha spectra (left) generated with low geometry alpha particle counting setup (right)

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SANDA – workshop on actinide target preparation and characterization – the need for radioanalytical chemistry

Abstracts

presented on 3rd day: 7 December 2023

CHARACTERIZATION OF ACTINIDE TARGETS

Klaus Eberhardt

Johannes Gutenberg-University (JGU), Research Reactor TRIGA Mainz

Actinide targets are widely used for different applications, e.g., in nuclear chemistry and -physics experiments, for the synthesis of the heaviest elements, for neutron absorption cross section measurements important for the development of advanced reactor concepts, and as neutron flux monitors and in neutron dosimetry. The targets must meet specific requirements of stability, areal density, homogeneity, and layer morphology and thus proper characterization of the actinide layer is of upmost importance for accurate measurements. This might include inspection of the targets prior to and also after irradiation experiments, which is of particular importance in heavy ion collision experiments at high beam intensities when target damage and thus actinide material loss can become an issue.

Characterization methods for thin actinide layers include a variety of techniques comprising such different methods as radiometric analysis, electron microscopy, x-ray spectroscopy and -diffraction, radiographic imaging, photoelectron spectroscopy, neutron activation analysis, optical microscopy, and atomic force microscopy. However, since the targets are radioactive not all methods are readily available to study radioactive samples. For on-line monitoring of target performance during irradiation and to detect beam induced transformations more sophisticated methods like elastic recoil detection analysis and rutherford backscattering spectrometry can be used.

This presentation comprises the techniques used at JGU that are readily available in a radiation protection area for the characterization of actinide layers on thin metallic backings. The following methods are routinely applied:

- α-particle spectroscopy, neutron activation analysis and γ-spectroscopy for determination of the areal density
- Radiographic imaging for layer homogeneity measurements
- Scanning electron microscopy for layer morphology studies
- Atomic force microscopy for determination of the surface roughness

X-ray photoelectron spectroscopy is also available but has not been used so far with actinide samples.

ELECTRON MICROSCOPY STUDIES ON NUCLEAR MATERIAL

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Imaging techniques play a pivotal role in material research, providing critical insights into the microstructural and compositional properties of materials. This is particularly relevant in the field of nuclear materials, where the understanding of these properties can significantly impact the efficiency and safety of the nuclear fuel cycle. This presentation will focus on the application of electron microscopy in the study of nuclear materials, with a particular emphasis on Scanning Electron Microscopy (SEM), Focused Ion Beam (FIB) microscopy, and Transmission Electron Microscopy (TEM).

The capabilities of Energy Dispersive X-ray Spectroscopy (EDS), Wavelength Dispersive X-ray Spectroscopy (WDS) and Electron Back-Scattered Diffraction (EBSD) will be highlighted, demonstrating their effectiveness in characterizing materials relevant to the nuclear fuel cycle. EDS (and WDS), with its ability to provide elemental composition, and EBSD, with its capacity to reveal crystallographic information, are powerful tools in the comprehensive analysis of nuclear materials. In the TEM, crystallographic information is provided by Electron Diffraction and additionally, elemental analyses, determination of chemical bonding, valence and conduction band electronic properties can be obtained from Electron Energy Loss Spectroscopy (EELS).

However, the application of these techniques in the nuclear field presents unique challenges, particularly in terms of safety and protection measures. The necessity of operating within gloveboxes and other protective environments can complicate the process, but it is a crucial aspect of ensuring safe and responsible research practices.

The presentation will also discuss the challenges and opportunities associated with the application of these techniques in the field of radioanalytical chemistry, particularly in the context of actinide target preparation and characterization. The ultimate goal is to underscore the importance of electron microscopy in advancing our understanding of nuclear materials, thereby contributing to the development of safer and more efficient nuclear technologies.

TARGETLABORATORY @ GSI AND FAIR

Birgit Kindler and Bettina Lommel

GSI Helmholtzcentre for Heavy Ion Research, Darmstadt, Germany

GSI operates a heavy ion accelerator facility for ions up to uranium up to 1 GeV/u. Currently we are building FAIR, Facility of Antiproton and Ion Researchone of the largest research projects worldwide.

The target laboratory is part of the scientific-technical infrastructure for research and accelerators.

Backings, foils and targets are provided over a wide thickness range from some 10 nms up to mms with a large variety of materials. We produce targets for experiments at GSI and for experiments with GSI participation at facility in Europe and abroad.

We apply PVD methods and mechanical procedures. The analytical methods available will be presented.

THE NEED FOR AN ACTINIDE LABORATORY

Ch. Stodel et al.

Grand Accélérateur National d'Ions Lourds (GANIL), Caen - France

With the new facility SPIRAL2 at GANIL, including high intensity beams from the superconducting linear accelerator (LINAG), the Neutron for Science experimental area (NFS) [1] and the "Super Separator Spectrometer" (S3) [2], cutting edge research in fundamental nuclear physics and applications is pursued. To achieve the envisaged extensive experimental program, specific research and developments work is being carried out on targets and their environment.

Actually, targets are an essential element in the experimental set-up as their properties contributes directly to the accuracy of obtained observables. A vast program concerns actinide targets, for which the availability of raw materials is scarce and, even more so, expertise in their manufacture and characterization is limited.

After introducing the facility, we propose to report on the achieved technical developments on rotating targets' stations and to focus on actinide targets. Considering their specification and need, a project for a dedicated actinide laboratory will be discussed.

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