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HoT in NF course for pilot on Moodle

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1.1	08/03/2023	Marko Štok	JSI part added, note on delay of 4 videos announced. Postponed to end of June 2023.
1.2	30/06/2023	Marko Štok	Videos ready, links uploaded in Moodle structure and information added to the deliverable.
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EXECUTIVE SUMMARY

The preparatory courses for a hands-on training in Nuclear Forensics were implemented as Moodle courses. The materials of the hands-on training have been prepared to the extent that the trainings could be conducted. LUH has already conducted the training with 6 students. The feedback was very good.

The courses are designed as a blended learning experiment. They offer a phase of preparation before the actual hands-on training in presence. The participants can get used to the topics in their own pace and a lot of time is saved onsite.

The courses are headed by LUH, JSI and CTU. Each Course has its own Moodle structure.

1. Analysis of HoT Particles and rapid detection of radiosilver (LUH)
2. Determination of U isotope ratios by MC-ICP-MS (JSI)
3. Alpha spectrometry of uranium isotopes (JSI)
4. AMS determination of U-236 isotopic ratio (CTU)

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PART 1 LUH – ANALYSIS OF HOT PARTICLES AND RAPID DETECTION OF RADIOSILVER

1 INTRODUCTION

Nuclear Forensics is the examination of nuclear and other radioactive materials using analytical techniques to determine the origin and history of this material in the context of law enforcement investigations or the assessment of nuclear security vulnerabilities (definition of the International Atomic Energy Agency (iaea.org)).

In this portion of the hands-on training, participants will learn about two important points in nuclear forensics: The fastest possible detection of radiosilver, and the examination of nuclear fuel particles. While the first is presented very directly in the course with instructions, the second is more of an introduction to the broad field of so-called hot particles.

The Moodle course is structured in such a way that the participants first have to go through a general quiz on the topic of radioactivity. They are encouraged not to cheat, as the test is primarily to give the teacher an idea of the student's knowledge level.

Gradually, they become familiar with concepts relevant to fuel particle research. In between each chapter there are small quizzes that ask the students to deepen their knowledge.

They receive basic instructions on how to handle the equipment they will later operate in presence.

The introduction to radiosilver is designed as a complete tutorial. The students start with the motivation and end with full instructions. Here, too, the students have to answer questions in between.

1.1 Structure of the Moodle course

The Moodle starts with a very short quiz to show this function to the students. One question is “Do you want to learn interesting things about Nuclear Forensics?”.

After that the students get redirected to the next chapter, the basic course about radioactivity. The students have to do a quiz, which they do not have to pass. It is for the teacher in order to get an understanding of their current knowledge. The main part of the questions will also return in the ending quiz, which the students actually need to pass to be allowed to work in the physical lab.

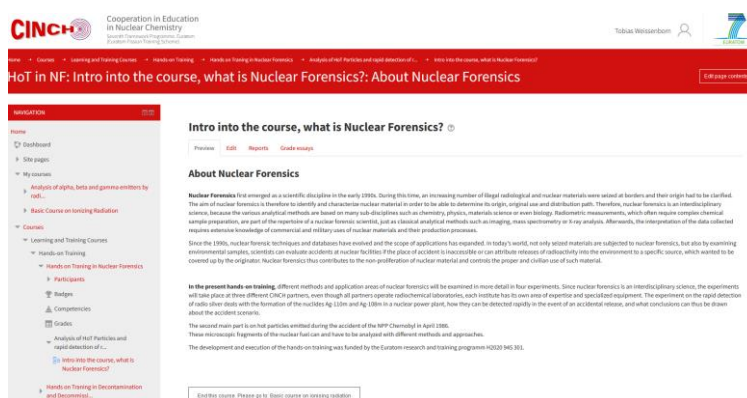


Figure 1 The intro to the course. A quick introduction into the topic of Nuclear Forensics and into the HoT.

The first learning chapter is about nuclear fuel fragments, so called “hot particles”. The students learn about their morphology, their origin, why they are a big concern and also about the different measurement techniques performed at the LUH.

Once profound with the theory, the course shifts to the methods performed at the HoT. The students learn about the practices concerned with the search for hot particles and how they are extracted. Once the particles are isolated on tungsten needles, targeted measurements can be performed on them. These measurements include gamma spectrometry, EDX and SIMS/SNMS measurements. The students learn about the SIMS system and why the isotopic ratios are important. The resonant laser SNMS is also explained briefly.

The chapter about the separation of selected radionuclides: radiosilver also starts with a bit of Theory. The students learn how the radiosilver is produced in the reactor and what the amount and ratio of radiosilver means for a theoretical accident. Also, the problem of a high background of gamma emitting radionuclides is introduced.

Before the students get to the actual experiment, they learn about the very complex decay schemes of ^{108m}Ag and ^{110m}Ag and the concept of auto deposition. The experimental part is explained on the next pages.

The course ends with a quiz, which the students have to pass. It contains some questions about the afore mentioned materials, but also repeats some of the questions of the first quiz. It is rounded with a few lab safety questions to ensure that students have sufficient prior knowledge.

1.2 Hands on Training in Hannover

The LUH decided to actually conduct the hands-on training at the IRS.

Six students from six different countries, from three different continents were selected out of over twenty applications. There was also a wide range of different levels of education. Three PhD students, two master's students and one bachelor's student.



Figure 2 The students in the laboratory with their supervisors Laura Leifermann and Tobias Weissenborn.

The Students arrived on Monday 29th in Hannover. The HoT started on the next day with a mandatory safety briefing after a small get-to-know game. The laboratory tour was very well received. The students had lunch together with the other employees of the IRS, where a friendly atmosphere between colleagues, students and supervisors could take place. Freshly strengthened the first work in the lab could be started.

The students were divided in teams. One group started with the production of tungsten needles for the particle extraction, while the other started with the preparations for the autodeposition of silver. After some time teams changed their tasks and one group was introduced to the SEM. At the end of the day, the first radiosilver samples could be put on the detector.



Figure 2 Students sample an asphalt core out of the city of Prypiat.

On the second day of the training, the work of the first was continued. One group had time on the SEM to measure their previously prepared samples, while the other started their own radiosilver preparations. On this day the practical course was supplemented by lecture on Mass Spectrometry by Dr. Michael Steppert of the IRS. As there was some spare time, Julia Stadler (IRS) held a spontaneous talk about her own PhD project in chemistry. Dr. Sergij Dubchak, an ukrainian scientist who just started to work at the IRS at that time, gave a talk about the Chornobyl accident with the focus on the current situation. The rest of the day was spent in the lab.

The last day of the training also had practical lessons in the lab and a shorter lecture on gas chromatography. The students managed to locate a hot particle during the lab time. They were also able to find it in the SEM and one particle was actually extracted onto a needle for further measurements. Sadly, there was not enough time left. The students also gave feedback. The students wished for more time to practise their newly acquired skills. The training could have been stretched without further work to four days, as simply more time within the existing agenda would have been needed.

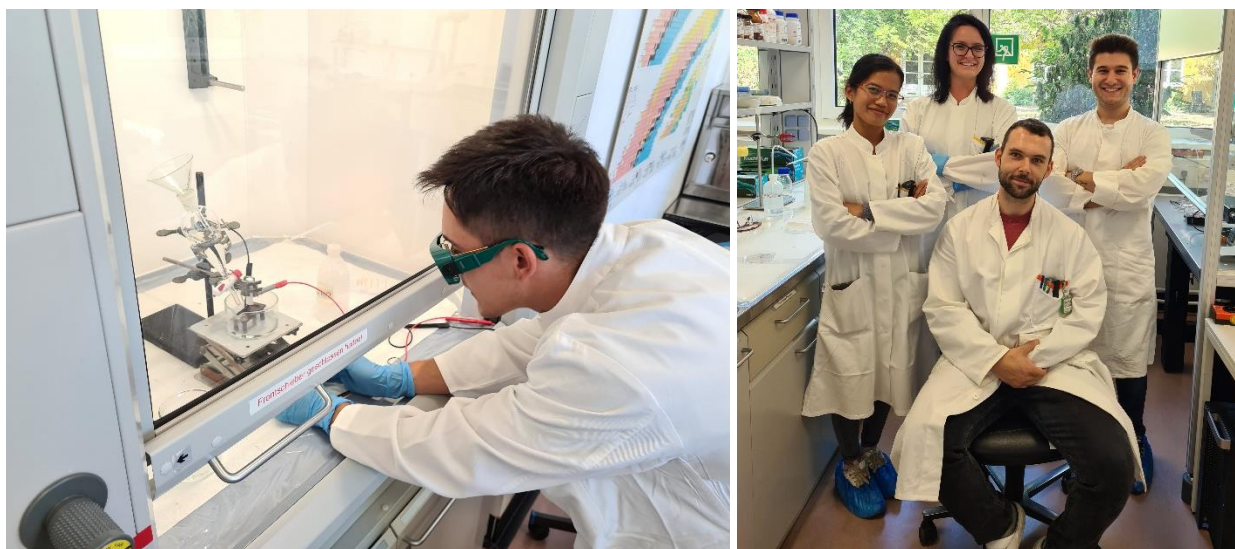


Figure 3 A student preparing tungsten needles and team red with their supervisor.

2 CONCLUSIONS

The Moodle course for the HoT in NF was created and the hands-on training itself was planned. LUH decided to try the course with actual students and the training was very well received by the diverse group. More time in the lab should be planned for further runs in a similar schedule.

PART 2 JSI – DETERMINATION OF URANIUM ISOTOPE RATIOS BY MC-ICP-MS AND DETERMINATION OF URANIUM ISOTOPES BY ALPHA SPECTROMETRY

1 INTRODUCTION

JSI has prepared two exercises for the HoT in nuclear forensics. Both are focused on determination of uranium isotopes, which is important signature in nuclear or other radioactive material. This signature can give us information whether or not U-235 is enriched or depleted in the sample. In addition, presence of U-236 can often indicate that such material has been irradiated, which is important information in revealing the history of nuclear material.

First exercise involves determination of U isotope ratios by MC-ICP-MS in swipe samples. Swipe samples are often taken in the case when we want to investigate non-disclosed presence of nuclear material. U isotope ratios can reveal us presence of enriched or depleted uranium, as well as presence of U-236. Second exercise involves determination of U isotopes by alpha-particle spectrometry. Similar to MC-ICP-MS, also this method can reveal presence of depleted or enriched uranium, however with less accuracy and precision. Nevertheless, alpha-particle spectrometry is more readily available in many labs compared to MC-ICP-MS.

Both exercises are time consuming and involve destruction of the sample, therefore they are normally applied in later stages of nuclear forensics investigation, following faster and non-destructive techniques. In addition, they require different skills, starting from selection of suitable sample preparation method, radionuclide separation techniques, usage of sophisticated measurement equipment and data analysis. The aim of the HoT is to equip participants with all necessary skills that they will understand the concepts in determination of uranium isotopes with MC-ICP-MS and alpha-particle spectrometry, as well as that they will be able to independently perform such analyses.

To reduce actual time spent on site during the presence phase of HoT, CINCH Moodle online platform (<https://moodle.cinch-project.eu/>) is used in order that participants get acquainted at their own pace with the information needed to effectively complete and participate in the HoT presence phase. Such blended learning approach has been found to be very effective in the past and substantially enhances user experience.

For the online part of both exercises, laboratory videos were produced and are showing step by step analytical procedure to be performed.

In the following chapters, Moodle online training part is presented in detail.

2 MC-ICP-MS DETERMINATION OF U ISOTOPE RATIOS

Moodle structure for the determination of U isotope ratios using MC-ICP-MS involves 10 laboratory videos and 4 quizzes, with which participants can test their understanding of the topic and newly acquired knowledge. Screenshot from the CINCH Moodle site is shown on Figure 4.

The screenshot shows a Moodle course page for 'MC-ICP-MS determination of U isotope ratios (JSI)'. The course is part of the 'Cooperation in Education in Nuclear Chemistry' program. The page features a sidebar with a course navigation menu and a main content area listing 10 video modules. Each module includes a video icon, a title, and a brief description.

Module Title	Description
Sample ashing	This video shows how to ash swipe sample for U isotope ratio analysis.
Acid digestion	In this video acid digestion of ashed swipe sample is presented.
Sample ashing and acid digestion	
UTEVA resin precleaning and conditioning	In this video precleaning and conditioning of UTEVA resin is shown.
Separation of uranium on UTEVA column	In this video you will learn how to separate uranium by using UTEVA column
Destruction of organic residue after UTEVA separation and dilution	In this video you will learn how to destroy organic residue from UTEVA resin separation and dilute sample for next steps.
Uranium separation using UTEVA column	
U measurements on ICP-MS	In this video you will learn how to measure U concentrations on ICP-MS.
Uranium measurements with ICP-MS	

Figure 4: Screenshot of CINCH Moodle materials for MC-ICP-MS determination of U isotope ratios.

Online part of the HoT introduces participant through all steps in determination of U isotope ratios by MC-ICP-MS. They involve sample ashing, where swipe sample is ashed at 650 °C. This is then followed by nitric acid digestion and preparation of the sample solution in media suitable to be used for uranium separation protocol. After that, participants can check their knowledge in quiz with eight questions.

Next set of videos introduce participant with necessary arrangements to separate uranium isotopes with the help of UTEVA extraction chromatography, as well as on how to prepare eluted uranium sample for the measurements. Also, here there is a quiz with seven questions where participants can check understanding of the topic.

Before we can measure and determine U isotope ratios on MC-ICP-MS, we need to know what is the U concentration in the sample that we can match it with standard. This is done by measuring of U concentration by ICP-MS, which is described in next video, followed by quiz with six questions.

Finally, series of videos introduce how to correctly set up and tune MC-ICP-MS to measure samples and determine uranium isotope ratios. Also, they are followed by the quiz with six questions.

3 ALPHA SPECTROMETRY OF URANIUM ISOTOPES

Moodle structure for the determination of U isotope ratios with alpha-particle spectrometry involves 11 laboratory videos, one lecture and 8 quizzes, with which participants can test their understanding of the topic and newly acquired knowledge.

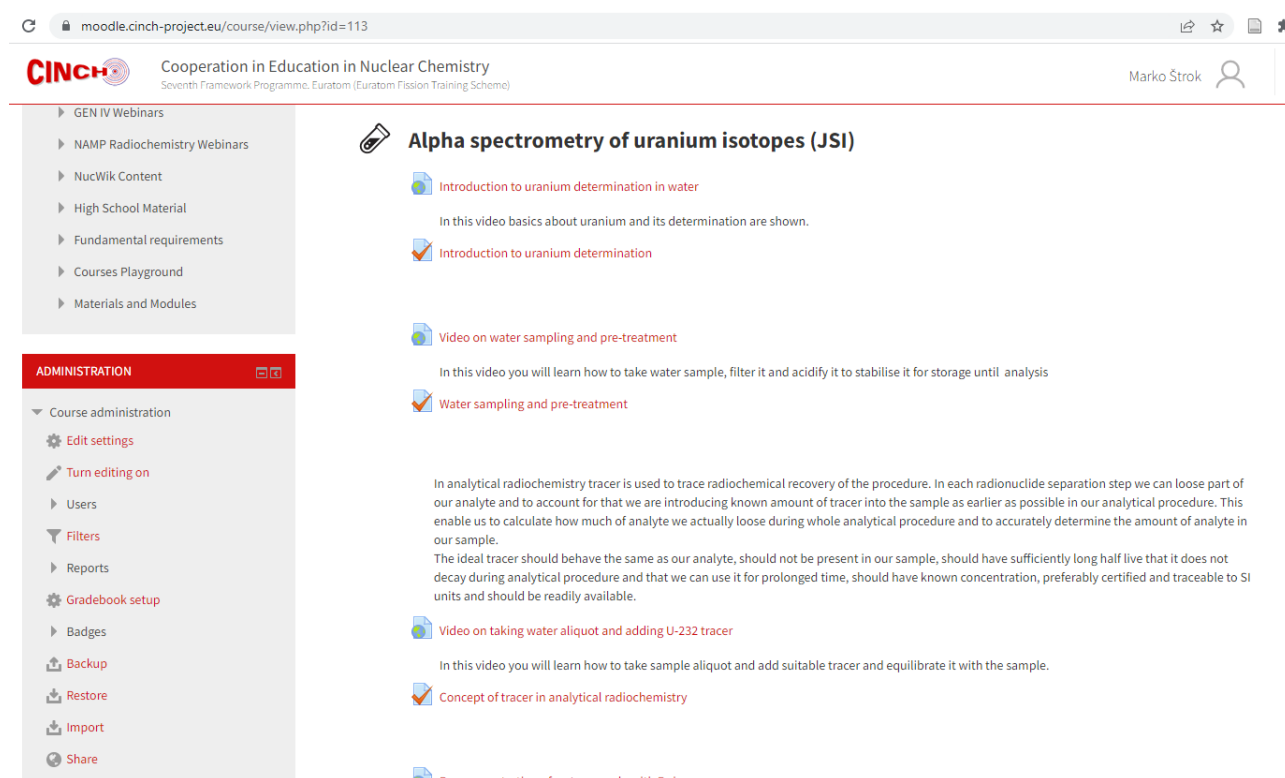


Figure 5: Screenshot of CINCH Moodle materials for determination of U isotopes with alpha-particle spectrometry.

Moodle online part of the HoT starts with the introductory video explaining uranium characteristics and is followed by quiz with six questions where participants can test their understanding of the topic. Next, water sampling and pre-treatment is presented, which is also followed by the quiz with six questions.

The participants are then familiarized with the concept of tracer in analytical radiochemistry, as well as how to take sample aliquot for analysis. This is then followed by another quiz with six questions where users can check their knowledge on the topic.

Next topic explained is pre-concentration of radionuclides from water sample, also followed by the quiz with six questions. Then, series of videos are showing how to separate uranium from other radionuclides using UTEVA resin, following quiz with six questions.

After effective radiochemical separation, next series of videos are showing preparation of counting source for alpha-particle spectrometry with micro-co-precipitation. Also, this topic is covered in the quiz with six questions.

Uranium measurements by alpha-particle spectrometry and calculation of results are covered in next sections, also including quiz with six questions enabling participants to check their knowledge.

Finally, lesson about quality assurance and quality control in analytical radiochemistry introduces participant to that important topic, which is also accompanied with the quiz with five questions.

4 CONCLUSIONS

Online part of JSI exercises for HoT on nuclear forensics hosted on CINCH Moodle enable participants to gain required background information before the presence phase and enable more efficient use of time and resources during presence phase. In addition, it allows participant to learn topics of interest at their own pace, which makes their learning experience better.

PART 3 CTU – ACCELERATOR MASS SPECTROMETRY DETERMINATION OF U-236 ISOTOPIC RATIO



1 INTRODUCTION

CTU prepared task complementary to the tasks about dealing with measurement of uranium isotopes and their ratios; this task is focused on measurement of ^{236}U as a radionuclide of both natural and mainly anthropogenic origin. This isotope belongs together with others to the so-called fingerprint of nuclear materials in nuclear forensics. On the other hands, this nuclide was introduced to nature by anthropogenic activities and plays a special role of the trace of natural processes, such as migration or other transport phenomena, namely in hydrosphere. Determination of ^{236}U at natural and close-to-natural concentrations is a complex task due to its abundance and specific activity.

Accelerator mass spectrometry (AMS) is an ultratrace analytical measurement method and its current development worldwide promises its further development. In addition, the number of AMS machines rises and AMS is becoming relatively available tool for routine analyses – namely of ^{14}C , but also for other long-lived trace nuclides such as ^{10}Be , ^{26}Al , ^{36}Cl , ^{41}Ca , ^{129}I , ^{236}U and other actinoids. It provides important information about isotopic ratios of these nuclides to their stable isotopes, thus allowing track various type of natural or anthropogenic processes.

This task should present students accelerator mass spectrometry as an analytical method on the edge of mass spectrometry and high energy beam physics, which is using chemical and physical aspects of the nuclide to suppress possible interferences. But more important, the role of contamination in the sample preparation is highlighted in the task, which plays crucial role in ^{236}U analysis both in natural and more in nuclear forensics field. Student will get knowledge and skills about uranium separation and experience about sample preparation, contamination and detection limits.

2 COURSE CONTENT AND STRUCTURE

This course consists of several main parts implemented to the excursion and laboratory work in the AMS laboratory. This is also represented in the Moodle structure in Fig. 6.



AMS determination of U-236 isotopic ratio (CTU)



Accelerator mass spectrometry - introduction

Presentation gives short overview about accelerator mass spectrometry principles and use. It is suitable to go through this presentation before studying the manual.



AMS fundamentals - outline

An outline of the presentation with commentaries, suitable for notes.



1 Determination of $^{236}\text{U}/^{238}\text{U}$ ratio in tap water with AMS

This task is dedicated to fundamentals of ^{236}U determination with accelerator mass spectrometry. It consists of simple separation steps, preparation of target matrix sample, target cathode, and measurement of $^{236}\text{U}/^{238}\text{U}$ ratio with AMS MILEA.



Overview about AMS task and U-236 measurement

The quiz aims on simple but comprehensive overview about AMS. It is recommended to start this quiz after finishing both theoretical and practical parts.

Figure 6: Screenshot of CINCH Moodle structure for determination of U isotope ratio with AMS.

2.1 Accelerator mass spectrometry – introduction

This is an introductory lecture presenting student fundamentals and brief history of AMS its principles and use. In addition to this lecture, its outline with commentaries and space for notes was created to allow student better follow and study in advance. This lecture will provide student with basic knowledge necessary for following excursion and laboratory task, on the other hand this is an advanced course and sufficient knowledge in (nuclear) chemistry and physics is required.

2.2 Excursion to the joint AMS laboratory

The excursion will take place in the joint AMS laboratory of CTU and Nuclear Physics Institute of the Czech Academy of Sciences, in Rez near Prague, inside institute property, just after the introductory lecture. Students will pass through laboratories and in the AMS hall, the machine and its principles will be demonstrated and explained.

2.3 Determination of ^{236}U in tap water

This task is dedicated to fundamentals of ^{236}U determination with accelerator mass spectrometry. Because it is not possible for students to manipulate with real nuclear forensics materials and evidences, the simply available source of samples was selected – tap water. Uranium concentrations in tap drinking water are usually well below drinking limits (depending on the country max 10 - 30

ppm) and this requires well managed pre-concentration and separation steps, because measurement of this nuclide at and above natural concentration requires sufficiently sensitive method and careful sample treatment and preparation.

2.3.1 Introduction about ^{236}U

Chemical and nuclear properties of ^{236}U are briefly presented as well as requirements to its reference materials.

2.3.2 Basic principles of uranium separation

Uranium as well as other actinides has relative complex chemistry and it results in selection of separation of pre-concentration methods. Some basics of using changes in uranium oxidation states, solubilities of complex formation are listed as well as application of the common separation principles and procedures - co-precipitation with non-isotopic carrier, ion exchange of carbonate complexes, and separation on UTEVA resin.

2.3.3 Fundamentals of accelerator mass spectrometry measurement and operation

Here, fundamentals of AMS principles and machine components are again summarized. This is included because in the practical part students will participate on tuning the machine – this crucial procedure requires also at least understanding of basic principles and knowledge of names, function, and effects of each of the AMS components. During tuning, student will learn again, how the machine is working.

2.3.4 Practical part

Practical part will take place in the laboratories. Students will follow tasks in the manual depending on the selected separation path given by the teachers – several options are listed. In general, practical work consists of the following subsequent parts – simple pre-concentration and separation steps, preparation of target matrix sample, preparation of target cathode, and measurement of $^{236}\text{U}/^{238}\text{U}$ ratio with AMS MILEA. At the end, student will calculate evaluate their result together with teachers.

2.4 Summarizing quiz

Final quiz about AMS principles, components, and use is prepared to help students to check their knowledge about the method.

3 CONCLUSIONS

This task aims on introducing a student to accelerator mass spectrometry analysis and also to preparation samples with trace content of the analyte. After this task, a student should be able to know procedures of ^{236}U analysis with AMS and their limits.



GENERAL CONCLUSIONS

Four complex tasks of trace and ultra-trace methods suitable for nuclear forensics were prepared. All together they will show students principles, requirements, and limits of these methods, so they will get knowledge about their application and should be able to decide, whether the method is suitable for particular problem.

One tasks - Analysis of HoT Particles and rapid detection of radiosilver, consisting of two subtasks – was successfully evaluated in the pilot run at LUH.