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THE NEW LABORATORY FOR HERITAGE SCIENCE AT MTA ATOMKI – PREPARING FOR THE PARTICIPATION IN E-RIHS



HERITAGE SCIENCE

MTA Atomki

Hungarian Academy of Sciences Institute for Nuclear Research Debrecen



ΜΤΑ ΑΤΟΜΚΙ Founded in 1954

HSLAB

The new Laboratory for Heritage Science at MTA Atomki - preparing for the participation in E-RIHS

The Institute for Nuclear Research (short name MTA Atomki) situated in Debrecen, is one of the member institutes in the research network of the Hungarian Academy of Sciences. The primary activity of MTA Atomki is devoted to microphysical research for the understanding of the laws of nature which contributes to the results of scientific research in the world and to the sustainability of the scientific and technological culture in Hungary.



The Laboratory for Heritage Science was founded to promote research in the field of cultural and natural heritage, in collaboration with national and international partners.

The Ion beam Applications group of the Laboratory of Ion Beam Physics applies atomic and nuclear physics for multidisciplinary research, such as the investigation of the composition of art and archaeological objects. The main activity of the C-14 group in the Isotope Climatology and Environmental Research Centre is radiocarbon dating. The HSLab project is a common endeavour of the Ion Beam Application group and the C-14 group to extend our competencies by introducing new techniques and acquiring new devices.

HERITAGE SCIENCE

is a relatively new term for the complex research on our cultural heritage, including treatment, conservation, interpretation and documentation. E-RIHS.hu is the Hungarian node of the European Infrastructure for Heritage Science (E-RIHS)

In addition to archaeology, museology, art history, anthropology and palaeontology, this field is increasingly dominated by analytical methods, including especially the almost non-destructive, physical procedures and their development. MTA Atomki has several decades of tradition in this research.

Heritage science is a dynamically developing area. The ESFRI Roadmap took up the European Research Infrastructure for Heritage Science (E-RIHS) initiative in March 2016, which unites high-quality European analytical facilities. laboratories and museums to create a unique pan-European research infrastructure.

As a result of the HSLab project, MTA Atomki aims to be one of the internationally recognized hubs of this multidisciplinary distributed research infrastructure.

In the preparatory phase Hungary is represented by the E-RIHS.hu consortium led by MTA Atomki, with the participation of the MTA Centre for Energy Research, the MTA Wigner Research Centre for Physics, and the Hungarian National Museum.

H2020 IPERION CH

One of the backgrounds of E-RIHS is the H2020 IPERION CH (Integrated Platform for the European Research Infrastructure ON Cultural Heritage) project which already represents a unique collaboration in the research of cultural heritage

The participating institutions offer access to their analytical equipment or databases for external users (*TNA: Trans-National Access*).

This activity is complemented by research tasks with the aim to improve the competencies of the infrastructures.

The Laboratory of Ion Beam Physics, MTA Atomki participates with elemental analysis in the TNA programme, while its main research task is the investigation of the effects of radiation in the case of sensitive materials. Compared to its role in IPERION CH, MTA Atomki will be represented more extensively in the E-RIHS infrastructure. This will be partly due to the new, state of the art imaging and analytical devices which will complement the currently applied, accelerator-based analytical techniques, and partly to the inclusion of our modern carbon dating facility into the European access scheme. Carbon dating, which is of extreme importance in archaeology, is offered through the accelerator mass spectrometer (AMS) operated, adhering to the highest standards, jointly by MTA Atomki and Isotoptech Ltd.







LABORATORY

EQUIPMENT PROCURED IN THE FRAMEWORK OF THE GINOP PROJECT

With the new analytical-imaging devices purchased in the framework of the GINOP programme, a world-class, complex instrument park is created which enables more complex examinations in a wide range of scales

The first step in the investigation of cultural heritage objects is optical imaging. For this purpose, a **digital 3D microscope** is being purchased which yields quantitative information about the structures of the object not only on horizontal but also on vertical scale. This is useful, for example, for the determination of the depth of carvings or height of reliefs.

The currently used accelerator based techniques for elemental analysis in MTA Atomki will be complemented with a **micro-XRF** device (*XRF: X-ray Fluorescence*); depending on the sample and the elements to be determined. In the case of cultural heritage objects, the non-destructive nature of the analytical technique is often crucial. Because of this, traditional electron microscopy, although it can give information even on nanoscale, is often not applicable. Therefore, the scanning electron microscope *(SEM)* to be purchased will operate with variable pressure, so it will be suitable for the investigation of vacuum-sensitive or insulator samples, too.

The instrumentation will also be fitted with a built-in Raman microscope, so it will function also as an integrated **SEM-Raman microscope.**

The analytical capabilities of the accelerator mass spectrometer (AMS) facility is augmented to meet the special requirements of archaeological finds which are currently excluded from the investigations

An **automated graphitisation system** will enable sample

preparation in a fast, clean, highly reproducible way in the case of organic archaeological finds. Collagen is removed from the bones during cremation, so the ashes cannot be measured with traditional AMS method. For this type of samples, inorganic bioapatite must be used for C-14 dating. For this purpose, a **carbonate handling system** is being purchased. In the case of bone samples, which are of extreme importance in archaeology, stable isotope measurements are also needed for the accurate results. The corresponding **special sample preparation system** has already been constructed for the laboratory.

ION BEAM ANALYTICAL GROUP

The currently applied methods in the laboratory are based on the physical interactions of energetic ions with matter

Depending on the type of interaction, the bombarding particles may scatter, decelerate, be absorbed in the sample while the atoms are excited and ionized or nuclear reactions occur.

These processes lead to electromagnetic radiation (optical, X-ray, gamma-ray) and / or the emission of particles, such as electrons, protons and alpha-particles.

The energy of the electromagnetic radiation and particles reaching the various detectors depends on the atoms or nuclei in the sample which are the sources of these emissions, which means on the elements in the investigated material, while the intensity is related to the concentration of these elements.

With these measurements, qualitative and quantitative information can be obtained about the elemental, in some cases chemical, composition of the sample.



THE MOST WIDESPREAD ION BEAM ANALYTICAL (IBA) TECHNIQUES:

- > Particle Induced X-ray Emission (PIXE): The most prevalent IBA technique in heritage science, it is based on the detection of the so called characteristic x-rays emitted by the atoms (through processes involving the electron shells). The energy of the radiation depends on the given element (atomic number). The method is applicable from magnesium or even carbon to uranium, depending on the detectors and the experimental set-up.
- Rutherford Backscattering Spectrometry (RBS): A portion of the bombarding ions, passing through the electron cloud, is scattered back from the nuclei, without changing the state of the target atom. The energy of the backscattered ion depends on the mass of the target nucleus.

The method is especially suitable to determine a compositional depth profile in the surface layers of the sample. RBS is used for the investigation of surface alterations, corrosion, and coatinos.

> Particle Induced Gamma-ray Emission (PIGE): Some of the incident ions interact with the target nuclei in a way that the collision results in a nuclear reaction. The consequent gamma or particle radiation gives information on the elemental (isotopic) composition of the sample. The PIGE method is suitable for the quantitative analysis of elements with low or medium atomic number (from lithium to sulphur), the sensitivity varies greatly depending on the element.

AMS C-14 GROUP



Dating based on the measurement of radiocarbon relies on the radioactive decay law, which states that the radioactivity of a given isotope in the sample decreases exponentially, at a rate specific to the isotope. For life on Earth, carbon is one of the most important elements. Its heavier, radioactiveisotope with the atomic mass number of 14 (⁽⁴C) occurs naturally. Free neutrons are created in the upper layers of the atmosphere in large quantities due to cosmic radiation.

The main route for the production of ¹⁴C is the nuclear reaction when a nitrogen atom absorbs a neutron, resulting in a radiocarbon atom.





¹⁴C, on the other hand, decays into nitrogen with atomic mass number of 14 (¹⁴N) with a half-life of 5,730±40 years. It is estimated that about 2 atoms of ¹⁴C are produced in every second for each square centimetre of the surface of the Earth. Radiocarbon is rapidly oxidized into carbon dioxide, thus labels the carbon dioxide in the atmosphere.



for AMS C-14 dating

Since the intensity of the cosmic radiation has been quite stable for a long time, and the half-life of ¹⁴C can be considered short in this respect, the cosmogenic ¹⁴C on the Earth is in equilibrium.

The specific activity of carbon in the formations created with the incorporation of carbon from the atmosphere follows the specific activity of the atmospheric carbon.

When the exchange with the environment is stopped, for example when the metabolism of a living organism is over as the organism dies, the uptake of ¹⁴C also ceases, and the concentration of ¹⁴C in the material decreases exponentially, according to the half-life. Radiocarbon dating has great importance in archaeology as the finds can be dated back to 50,000 years.

In MTA Atomki, the method was introduced in the 1980s, since then we have seen an increasing number of requests from both Hungary and abroad.

SAMPLE TYPE	CARBON CONTENT (%)	REQUIRED SAMPLE MASS FOR AMS C-14 DATING
charcoal, peat	50-90	1-50 mg
wood	2-10	10-125 mg
fabric, canvas	10-50	2-25 mg
sediment, soil	0.2-5	20 mg -1 g
bone, tooth	1-5	0.2 – 3 g
coral, dripstone	10	25 mg
groundwater, aquiferic water	0.01	50-200 ml
air	0.04	2-3 litres

The gas proportional beta counting (GPC) method is rather slow, therefore meeting the demands for measurements became more and more difficult.

A new C-14 laboratory, based on the accelerator mass spectrometry (AMS) technique was begun in the institute in 2007. A dedicated sample preparation laboratory was established and a MICADAS type mass spectrometer with enhanced gas ion source was developed in collaboration with the Ion Beam Analytical Laboratory of ETHZ (Zurich), Switzerland. This compact, specialized system installed in 2011 was the third of this kind worldwide. Prof A. J. Timothy Jull, a renowned researcher from the carbon dating facility at the University of Arizona also joined the team, leading the efforts to utilize all the unique possibilities offered by the new equipment.

In addition to the significant infrastructural expansion in the recent years, new methodologies and devices have also been continuously developed.



ICP-MS LABORATORY

ATOMIC SPECTROSCOPY WITH HIGH SENSITIVITY

The Agilent 8800 inductively coupled plasma-mass spectrometer (ICP-MS) equipped with collision / reaction cell can detect elements in trace amounts.

It is also suitable to determine isotopic ratios. The collision

cell and the tandem mass spectrometer configuration allows measurements without interferences. The sampling system was complemented with a laser ablation device in 2015, creating the opportunity to analyse cultural heritage objects, as well.

PARTNERSHIP

INVESTIGATION OF BRONZE AGE GOLD OBJECTS IN THE FRAME OF THE E-RIHS.HU PLATFORM

Although gold jewellery coming from treasure troves, graves, or less often from settlements are mainly interpreted as prestigious objects, they are also important from the archaeological point of view, indicating the possible sources of the material

The primary sources of the prehistoric European gold jewellery are the gold ores in the mountains while the secondary sources are the alluvial deposits – deposits in stream beds. According to the current understanding, mining of the gold only began in the Iron Age. To trace the social exchanges intensified during the Bronze Age (between 2600/2500 and 800 BCE), it is important to know the

sources of the raw materials used by the craftsmen of the era. In collaboration with the Momentum Mobility Research Group of the Research Centre for the Humanities, Hungarian Academy of Sciences (Budapest), the Hungarian National Museum (Budapest), and the Déri Museum (Debrecen) the composition of gold objects held in Hungarian museums were investigated using non-destructive PIXE analysis in MTA Atomki.





100 μm x 100 μm

Elemental maps on the inhomogeneous sample (ChertPIXE / Marta Sánchez de la Torre)

CHERT FROM THE PYRENEES - IPERION CH PROJECT

In the case of minerals, it is important to distinguish between the matrix of the sample and the inclusions. By scanning the ion beam, elemental maps can be produced

In the frame of the IPERION CH EU H2020 project, Palaeolithic chert artefacts, as well as geological samples from chert outcrops from the Pyrenees were investigated.

By using the ion microprobe, not only the concentration but also the distribution of elements can be determined. Specific areas of interest can be selected on the elemental maps where further measurements are carried out.

Trace elements found in the matrix and in the inclusions of the samples point to the possible raw material sources for the artefacts.



PARTNERSHIP

THE REMAINS OF SAINT ASTRIK WERE IDENTIFIED BY AMS RADIOCARBON DATING AT MTA ATOMKI

Astrik, who brought the crown (known as the Holy Crown of Hungary) to King Stephen I, later became the first archbishop of Kalocsa. The identification of his relics was announced on the website of the Diocese of Kalocsa-Kecskemét

Previously it was assumed that the red marble sarcophagus found in 1911 belonged to Saul from the family of Győr, the archbishop who died in the beginning of the 13th century.

Painting depicting Astrik, archbishop of Kalocsa

(Archiepiscopal Treasury, Kalocsa)

samples from a finger bone, researchers at MTA Atomki and Isotoptech Ltd. announced that the radiocarbon measurements defined the date of death of the archbishop buried in the grave at 1001–1030 AD. This coincides with the scanty historical data on Astrik.

In April 2014, after examining

Although radiocarbon dating has been carried out with great precision for decades in MTA Atomki, to perform this measurement in the past one would have needed at least 200g bone sample from the precious finding.

Thankfully, by using the most modern accelerator mass spectrometric technique, 2g material from the phalanges was sufficient for the radiocarbon dating.

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from the measurements, using the calibration curve



Phalanges of the archbishop, used for the AMS radiocarbon measurement

The previously used, traditional radiometric techniques (liquid scintillation counter: LSC, gas proportional counter: GPC) are based on the measurement of radioactivity. By contrast, the AMS mass spectrometric method determines the ¹⁴C/¹²C isotopic ratio directly.

The isotopic ratio is in the order of magnitude of 10⁻¹² for the currently living organisms, while in a 50,000 year old sample, it could have decreased to one-thousandth of this. The maximum age which can be determined by this method is about 50-60 thousand years.



Chemical pre-treatment of a bone sample in a closed reaction cell at the AMS sample preparation laboratory. Debrecen