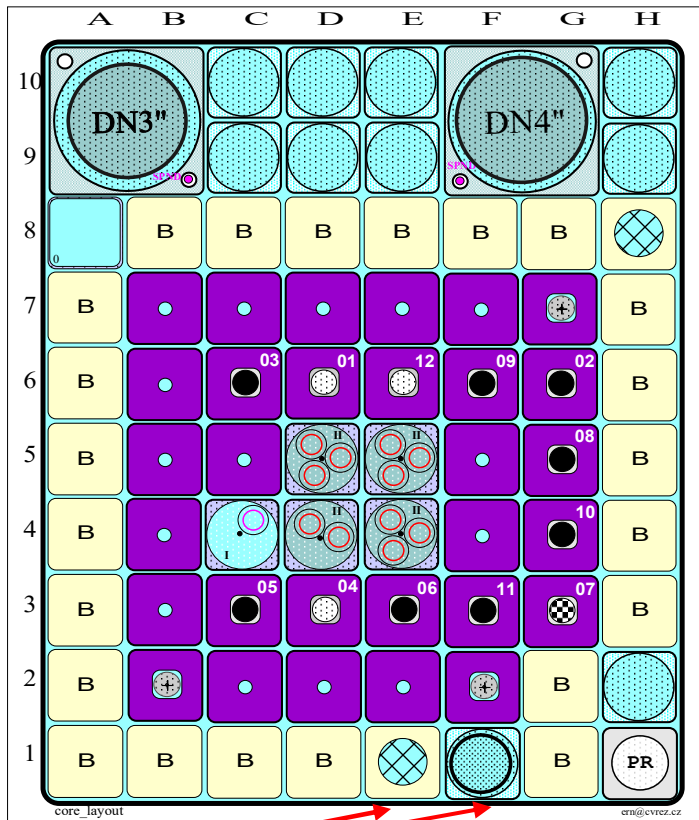


**Center of Accelerators
and Nuclear Analytical Methods
(CANAM)**

Neutron Physics Laboratory

Neutron Activation Analysis (NAA)

Neutron activation analysis (NAA) at vertical channels of the LVR-15 experimental reactor



H8 - long-time irradiation

H6 - long-time irradiation, formerly

H5 - long-time irradiation, formerly

H1 - short-time irradiation with pneumatic facility (transport time ~ 3.5 s)

E1, F1 - long-time irradiation, present







-  fuel
-  beryllium
-  facility for Si doping
-  facility for U irradiation
-  facility for Ir irradiation
-  channels for NAA irradiation

Fig. 1. Example of active core configuration

Neutron fluence rates of up to $5 \cdot 10^{13} \text{ cm}^{-1} \text{ s}^{-1}$ are available in vertical channels H1, H5, H6, H8. Epicadmium irradiation possible in channels H1 and F1.

NAA modes and standardization



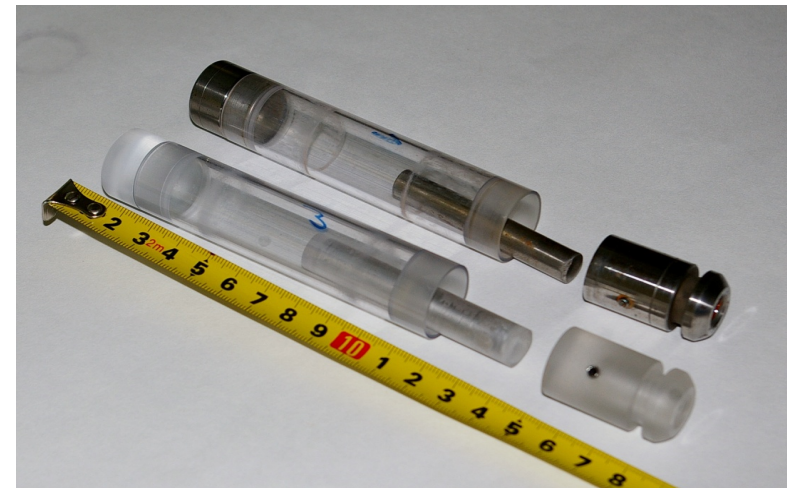
- Using both short-time (10 s - 3 min.) and long-time (several hours - several days) irradiations, information about concentrations of up to **65 elements** can be obtained, in many cases by non-destructive, so-called instrumental neutron activation analysis – **INAA** with **detection limits down to sub- $\mu\text{g g}^{-1}$** for many elements
- Epithermal instrumental neutron activation analysis – **EINAA** yields **improvement of detection limits for selected elements** up to one order of magnitude compared with INAA
- Procedures for radiochemical neutron activation analysis – **RNAA** are available for the elements V, Cr, Co, Ni, Cu, As, Se, Mo, Sb, I, rare earth elements, Re, and Hg that yield **detection limits down to the ng g^{-1}** level
- Both **relative and k_0 standardization** using KAYZERO for Windows and k0-IAEA software available

New facility



Device for cryogenic milling at temperature of liquid nitrogen Freezer-Mill 6770

- Extremely useful for milling and homogenization of materials difficult to desintegrate and homogenize by conventional milling





Examples of NAA projects within the CANAM infrastructure in 2013-2015

Methodological research in k_0 standardization



- Following the development of new neutron flux monitors consisting of the elements Au+Mn+Rb for short-time irradiation (Kubešová et al. 2011), we designed and validated a new monitor set consisting of the elements Au+Mo+Rb for long-time irradiation

M. Kubešová, I. Krausová, J. Kučera, J. Radioanal. Nucl. Chem., 300 (2014) 473-480.

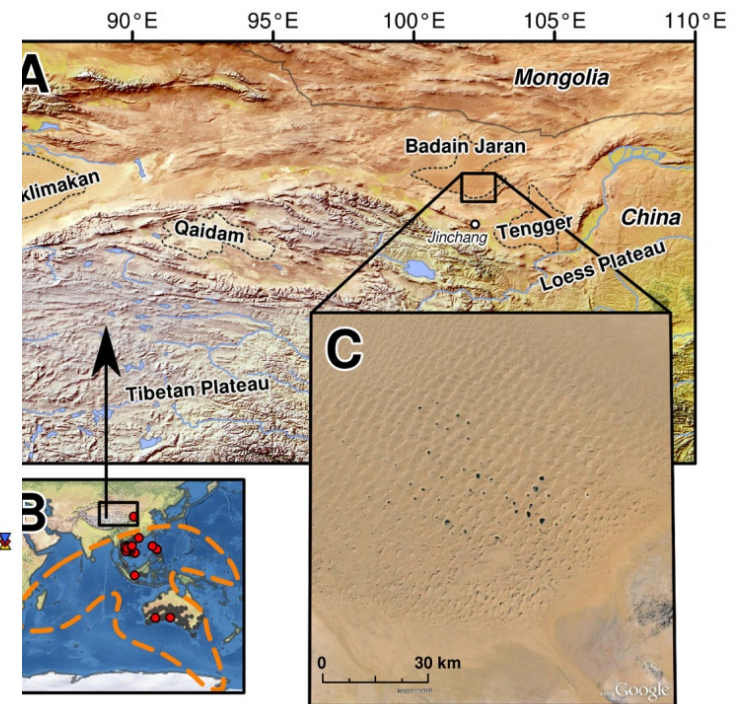
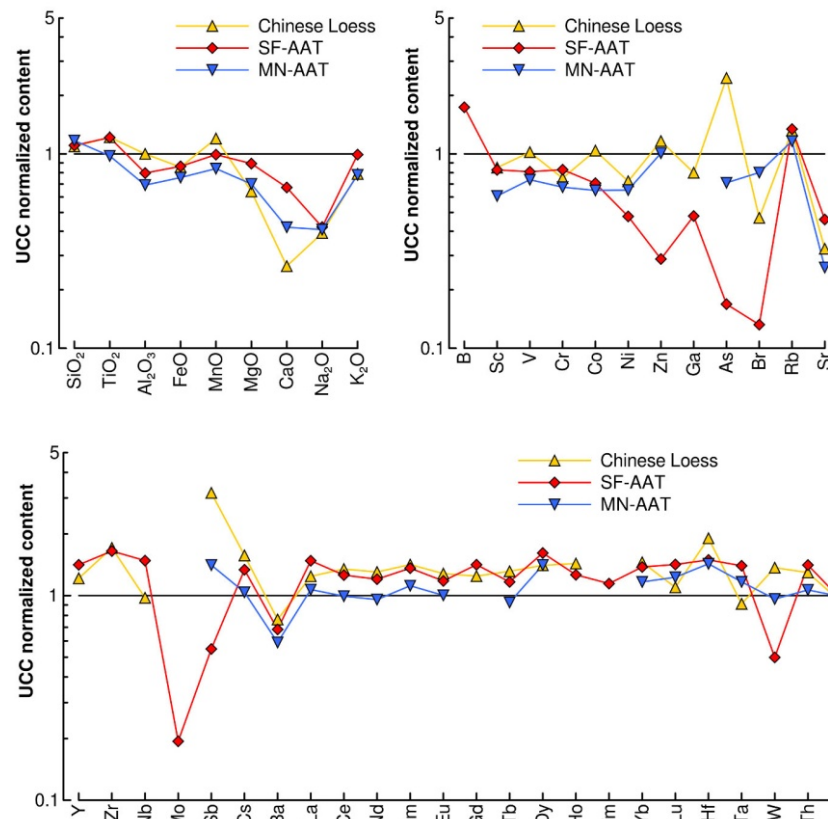
Geochemical research



- Anomalous uranium mineralization in coal from Odeř in the northernmost part of the Sokolov Basin, Czech Republic (uranium contents ranging from 0.02 to 6 wt.%) was studied using various analytical methods, including INAA

M. Havelcová, V. Machovič, J. Mizera et al., *J. Environ. Radioact.*, 137 (2014) 52-63.

- New location of the parent crater of Australasian tektites (AAT) has been proposed based on analysis of AAT samples by INAA and IPAA, and literature geochemical data.



J. Mizera, Z. Řanda, J. Kameník, *Earth Science Reviews*, 2015 (accepted)

Mycogeochemical research



After previous finding a macromycete (mushroom fruiting body), which is a hyper-accumulator of Ag, a new species of the subgenus *Phlegmacium* - *Cortinarius prodigosus* has been reported from the Czech Republic and Hungary, and assayed by INAA.



Amanita strobiliformis

~ 1250 mg Ag/kg (d.m.)

disregarding the composition
of the substrate



Cortinarius prodigosus

No special accumulation abilities - trace element contents range from 0.0x to xxx mg kg⁻¹ d.m.), of the macro-elements K is the highest - 4.14 ± 0.04 % d.m.

Environmental studies



- Comparative determination of Hg in contaminated soils was performed by RNAA and AAS for quality control purposes.

J. Sysalová, J. Kučera, M. Fikrle, B. Drtinová, *Microchem. J.*, 110 (2013) 691–694.

- Lignite samples from the Kışlaköy open cast mine in the south eastern Turkey were characterized by k_0 -NAA with emphasis on the potentially hazardous elements, such as As, Cd, Co, Cr, Mn, Ni, S, Sb, U.



M. Kubešová, E. Orucoglu, S. Erenturk et al., *J. Radioanal. Nucl. Chem*, 2015, DOI 10.1007/s10967-015-4550-2

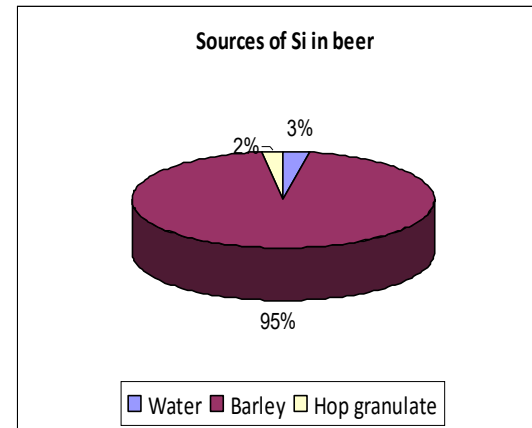
- Contents of 26 elements were determined by k_0 -INAA in soils and tree leaves collected in Istanbul for biomonitoring purposes. Elevated As and Sb contents were found in soils, whereas high Mn contents were found in tree leaves.

A.N. Esen, M. Kubešová, S. Hacıyakupoglu, J. Kučera, *J. Radioanal. Nucl. Chem*, 2015, submitted

Agriculture and nutrition



- Impact of the brewing process on the concentration of silicon (may be an important factor in preventing Alzheimer's disease) in lager beer was studied by INAA.



I. Krausová, R. Cejnar, J. Kučera, P. Dostálek, J. Inst. Brew. 120 (2014) 433–437.

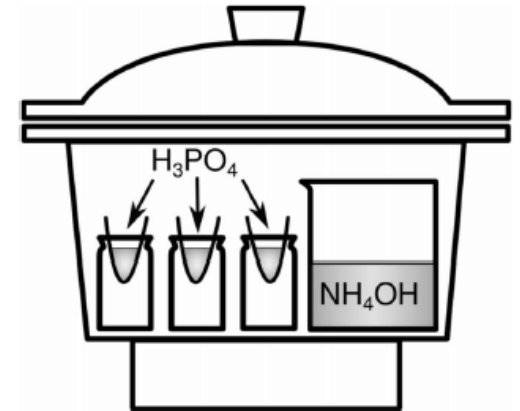
- The ability of bread and durum wheat to accumulate Se via a soil-addition procedure was studied by RNAA.

C. Galinha, A.M.G. Pacheco, M. do Carmo Freitas, M. Fikrle, J. Kučera et al., J. Radioanal. Nucl. Chem. 304 (2015) 139-143.

Material science and reference materials

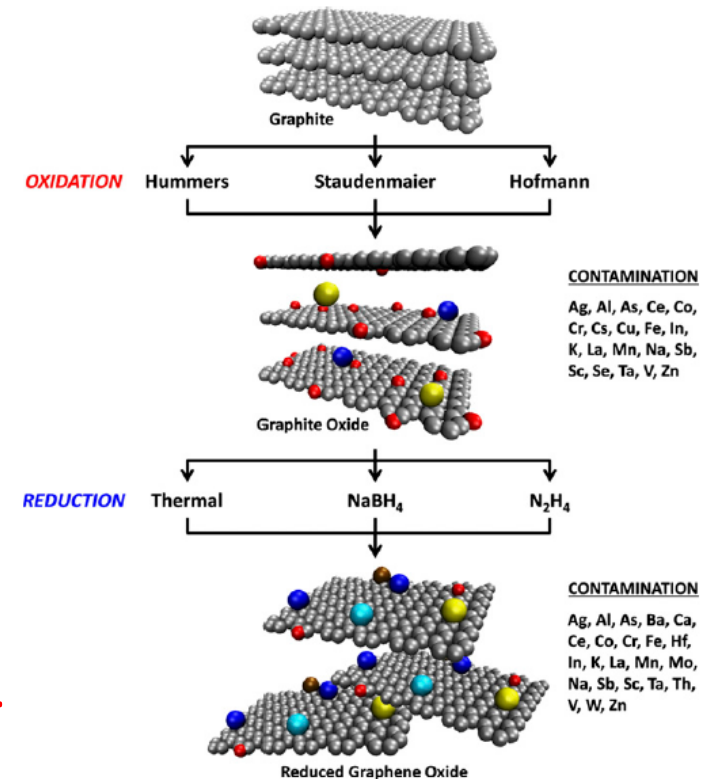


- Novel, simple and contamination-free procedure for the transformation of H_3PO_4 into $(\text{NH}_4)_3\text{PO}_4$ has been developed, which is based on isothermal distillation of ammonia vapours. The „solidified“ H_3PO_4 can be analyzed by INAA without any problem.



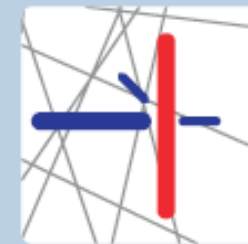
J. Kameník, H. Amsil, J. Kučera, J. Radioanal. Nucl. Chem., 304 (2015) 157-162.

- Using k_0 -NAA we showed that different oxidation and reduction steps introduce varying types and amounts of elements into the graphene materials. These metallic impurities are able to alter the graphene materials' electrochemical properties significantly and have wide-reaching implications on the potential applications of graphene materials.

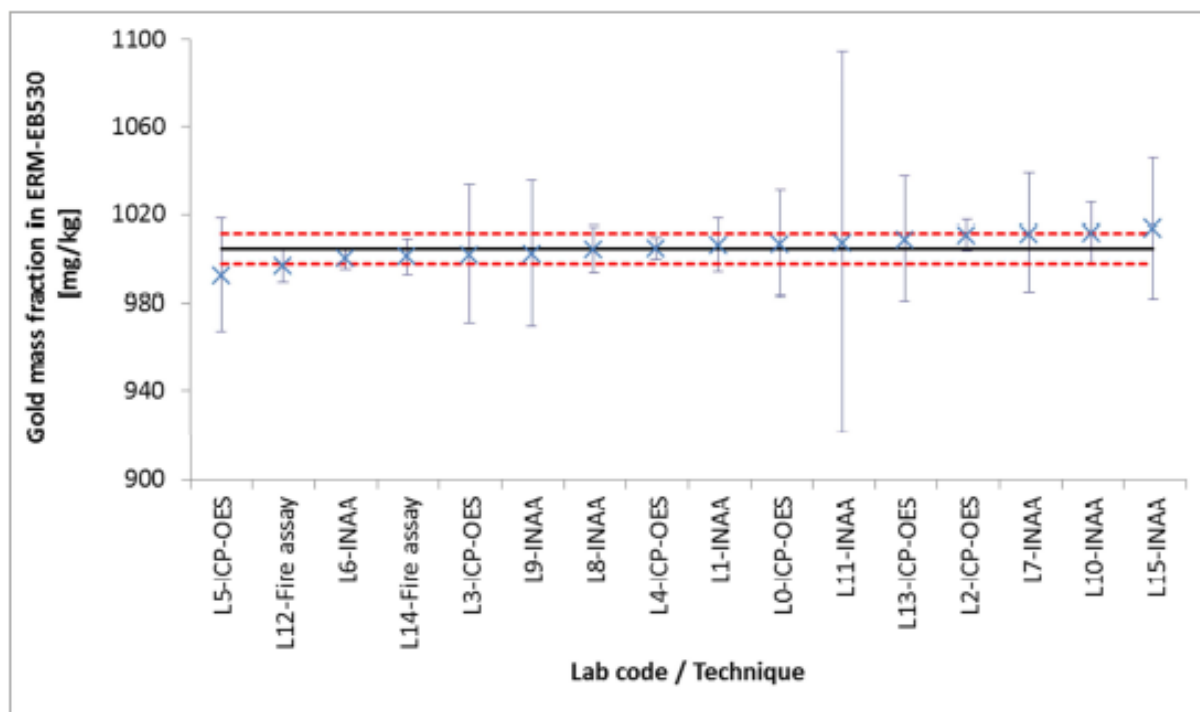


C.H. An Wong, Z. Sofer, M. Kubešová, J. Kučera et al., Proc. Nat. Acad. Sci. USA 111 (2014) 13774 – 13779.

Material science and reference materials



- We participated in an IRMM organized intercomparison among laboratories of demonstrated competence and adhering to ISO/IEC 17025 aimed at certification of the gold mass fraction in the new Al-0.1%Au alloy: ERM[®]-EB530



Our gold mass fraction value of $1001 \pm 5 \text{ mg kg}^{-1}$ obtained by INAA has been used for deriving the certified value of $1007 \pm 7 \text{ mg kg}^{-1}$

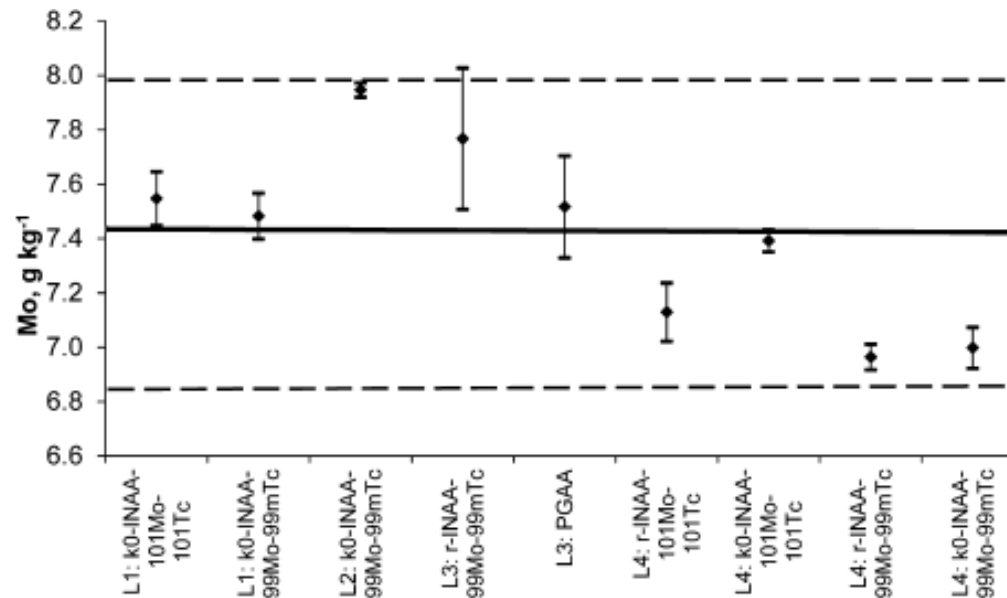
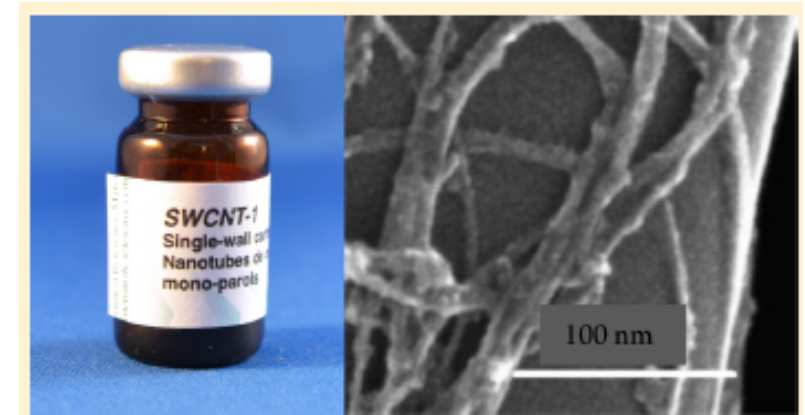
Figure E1. Mean gold mass fraction in ERM-EB530 reported by participating laboratories. Error bars represent expanded uncertainties as reported by participating laboratories. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code and technique used.

J. Kučera, J. Kameník, in T. Bacquart et al., Certification report, The certification of the gold mass fraction in Al-0.1%Au alloy: ERM[®]-EB530, IRMM, Geel, 2014, EU 26830 EN

Material science and reference materials



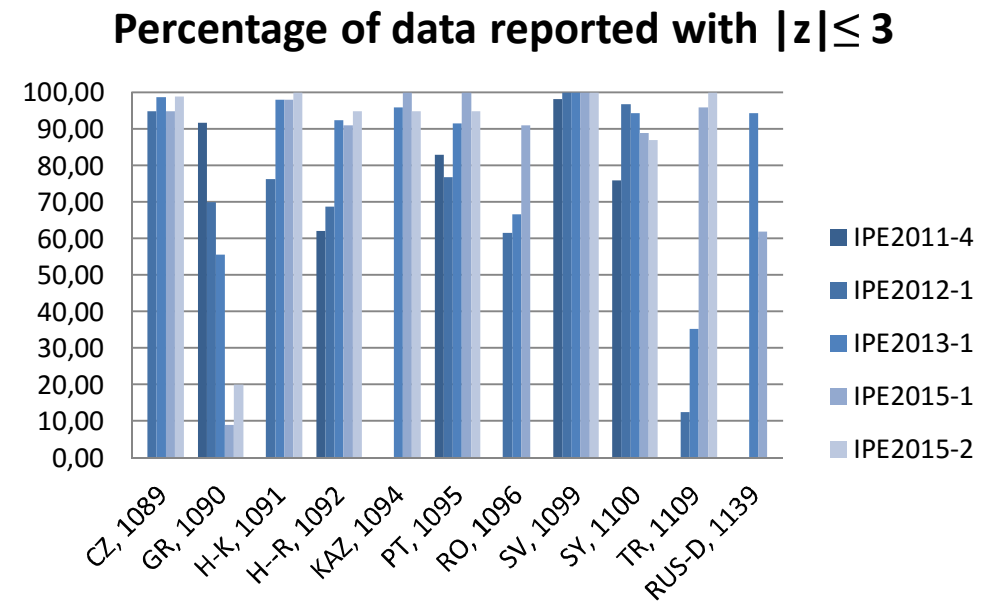
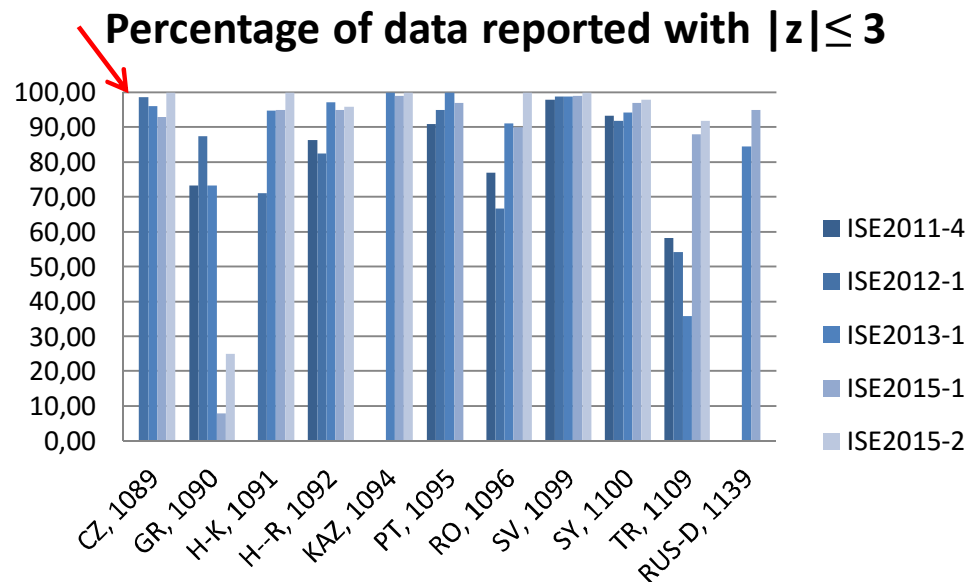
- We were among four leading NAA laboratories (NIST, ANSTO, CENA USP) that using INAA determined values in agreement with National Research Council Canada (NRCC) for certified values of Fe, Co, Ni, and Mo in CRM Single Wall Carbon Nanotubes (SWCNT-1) and provided mass fraction values for 13 additional elements, namely, Na, Mg, Al, K, Ca, Ti, V, Cr, Mn, Br, La, W, and Au, which were assigned as reference values.



Material science and reference materials



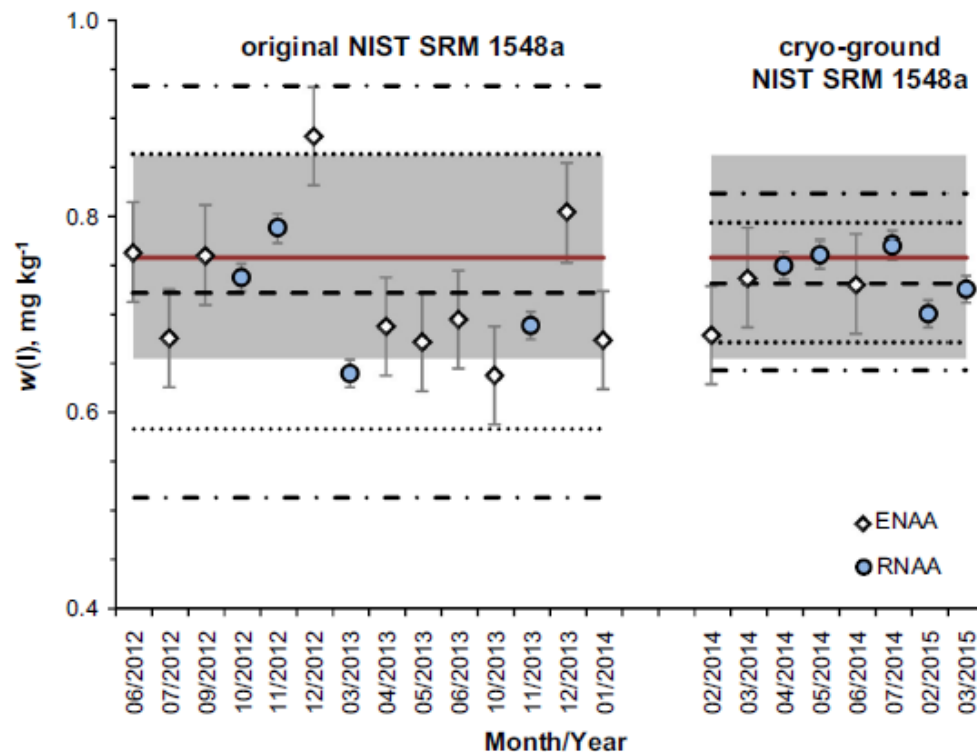
- In 2011 – 2015, we successfully participated in six rounds of proficiency tests using k_0 -NAA under the coordination of the IAEA. In each round four plant and four soil materials provided by WEPAL were analyzed and the results obtained were evaluated using z-scores. About 30 laboratories from Europe, Asia, and Latin America took part in this quality control exercise.



Material science and reference materials



- NIST SRM 1548a Typical Diet has a certified iodine mass fraction value of 0.759 mg kg^{-1} , with a rather large expanded uncertainty (relative value 13.6 %). We have proven that cryogenic grinding of NIST SRM 1548a (at the temperature of liquid nitrogen) yields a material with the standard deviation due to an inhomogeneity that is lower (by a factor of 3) compared with the original material. Thus, we have obtained a quality control material that sets up more stringent requirements on the validation and quality control procedures.



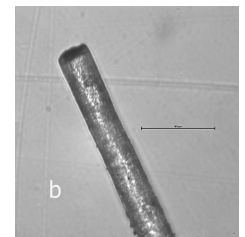
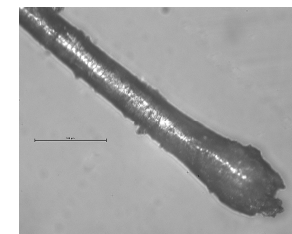
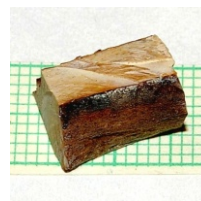
J. Kučera, J. Kameník, *Accred. Qual. Assur.*, 20 (2015) 189-194.

Cultural heritage

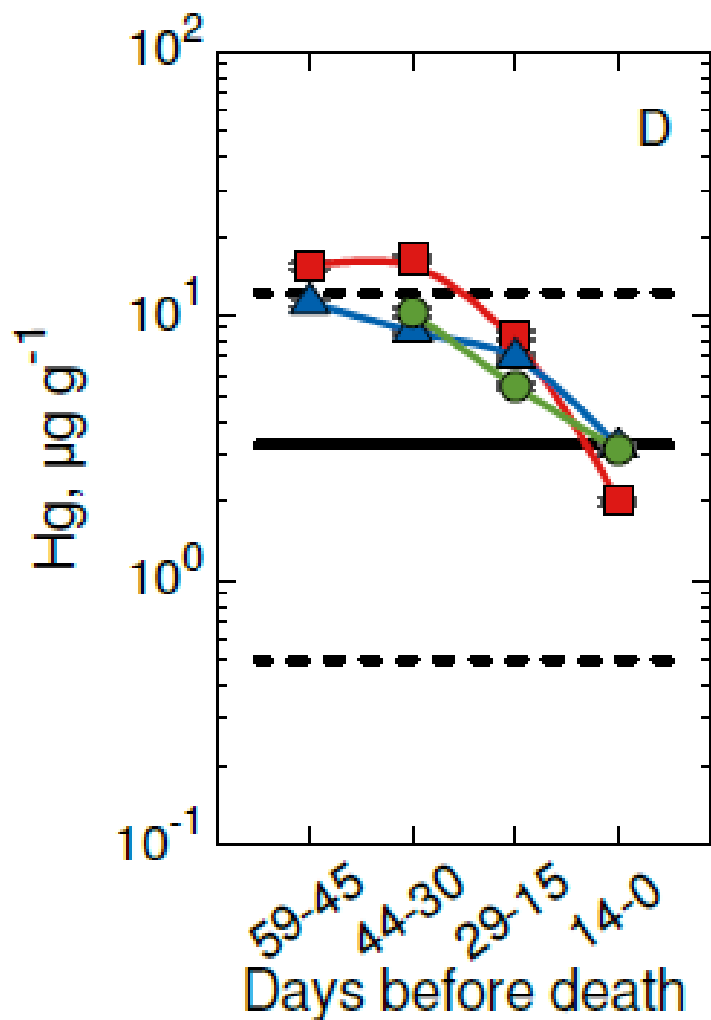
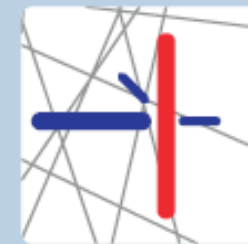


NAA of remains of Tycho Brahe (1546-1601)

After re-opening of the Tycho Brahe's tomb in Prague in 2010 by a Czech-Danish consortium, samples of hair and bones were procured and analyzed by NAA and μ -PIXE in Řež and by AAS in Odense to find out whether the world renowned astronomer was poisoned by mercury as it was rumoured.



Cultural heritage

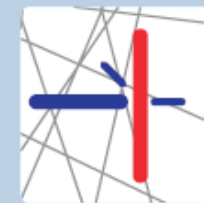


Determination of Hg by **RNAA** of 5-mm hair segments proved that acute exposure (**poisoning**) of Tycho Brahe can be excluded and determination of Hg by AAS and RNAA in bones proved that long-term Hg exposure can be excluded, as well.

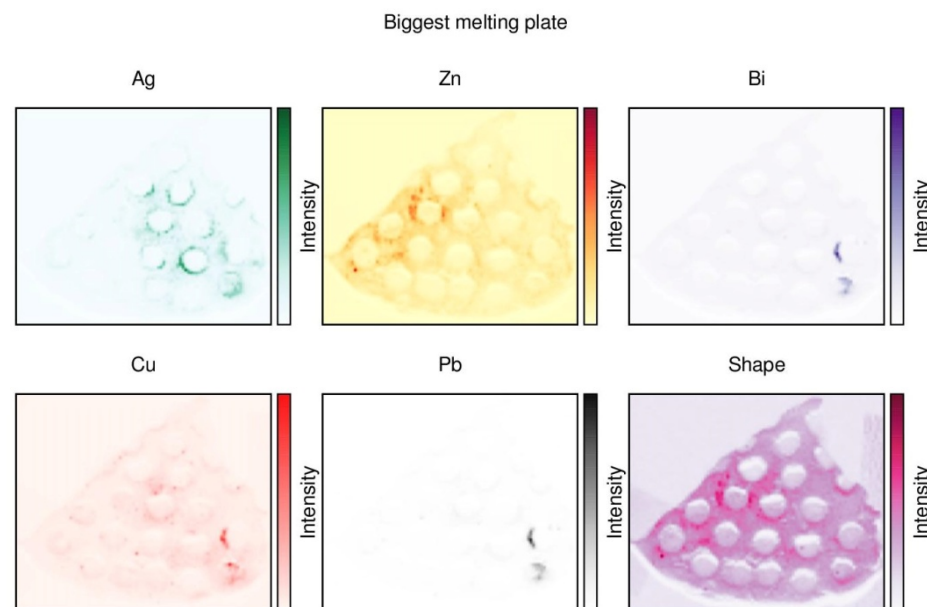
Toxicological significant Hg level 200-800 $\mu\text{g g}^{-1}$

K. L. Rasmussen, J. Kučera, L. Skytte, J. Kameník, V. Havránek, et al., *Archaeometry*, 55 (2013) 1187–1195.

Cultural heritage (XRF)



For analysis of Celtic melting plates from Stradonice (16pcs.) an innovative method - raster scanning XRF - was used. Although this method gives us only qualitative results they are extremely valuable from the archaeological point of view. The processed data of melting plate are unique, at least on the territory of Central Europe.



M. Fikrle, in J. Militký, Hradiště oppidum at Stradonice. Commented catalogue of coin findings and documents of coinage. Abalon, Ltd., Prague, 2015, 735 pp.

Future plans

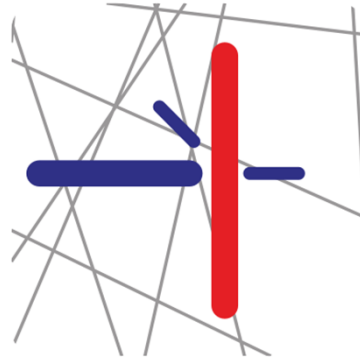


Accelerator mass spectrometry with a 1-MV machine for determination of long-lived radionuclides, such as ^{14}C (radiocarbon dating, environmental studies) ^{10}B , ^{26}Al (geochemistry, global climate changes) ^{99}Tc , ^{129}I and selected transactinides (radioecology, nuclear forensics)



HVE 1,0 MV Tandetron ME AMS System, Total System

Cooperation with the Czech Technical University (Prof. Jan John) within the Operational Programme Research, Development, Education (MEYS) - estimated cost 16 M€



Thank you for your attention!

Presented by Jan Kučera
Head of Neutron Activation Analysis