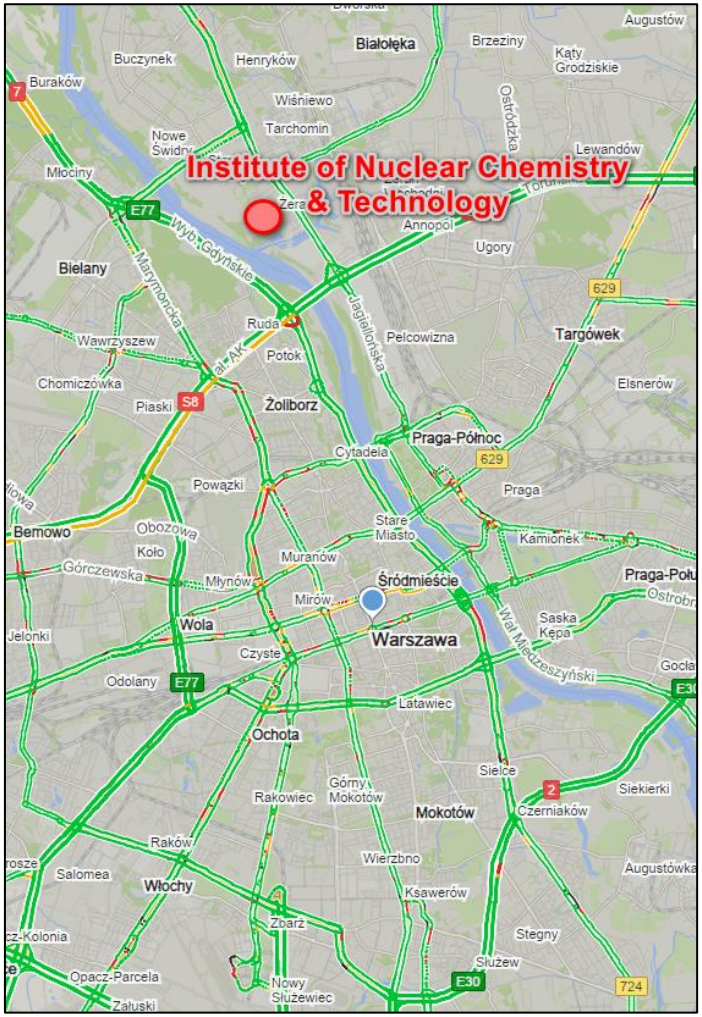
DETERMINATION OF TRACE ELEMENTS IN FOOD AND THE ENVIRONMENTAL SAMPLES.

P. Kalbarczyk[1], L. Fuks[2], E. Chajduk[1], H. Polkowska-Motrenko[1]

[1] Laboratory of Nuclear Analytical Methods, Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warszawa, Poland
[2] Centre for Radiochemistry and Nuclear Chemistry, Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warszawa, Poland

E-mail: P.Kalbarczyk@ichtj.waw.pl



The **Laboratory of Nuclear Analytical Methods of the Institute of Nuclear Chemistry and Technology** specializes in nuclear and nuclear-related analytical methods for applications in many specific fields, among other health and environmental problems.

The main area of activity of the Laboratory includes inorganic trace analysis. For this purpose, new procedures of chemical analyses for various types of materials have been developed.

The main analytical techniques, but not the only, employed in the Laboratory comprise: neutron activation analysis with the use of a nuclear reactor (instrumental and radiochemical modes), inductively coupled plasma mass spectrometry (together with laser ablation and HPLC) as well as gamma-ray spectrometry and alpha- and beta-ray counting.

Gamma neutron activation analysis (NAA) is a technique used for determining the presence and amount of many elements simultaneously in samples ranging in size from small to very large. The sample is continuously irradiated with a beam of neutrons The elements of the sample absorb these neutrons and emit prompt gamma rays which are measured with a spectrometer. Each element emits a unique gamma ray. The energies of these gamma rays identify the neutron-capturing elements, and the intensities of the peaks at these energies reveal their concentrations.

Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) uses a nanosecond-pulsed laser beam to remove material from the surface of a sample. The interaction of the laser and the sample surface causes heating, evaporation and ionization of sample material, in a process called “laser ablation”.

A plume of particles and ions are generated, and then carried via a constant flow of argon and/or helium gas to an ICP-MS. The sample material is subsequently ionized in an inductively coupled plasma, and its atomic species are transported as ions, and are separated and analyzed based on their mass-to-charge ratio over time.

LA-ICP-MS can therefore provide major and trace element compositions in a sample down to detection limits of 10’s of parts-per-billion (ppb).

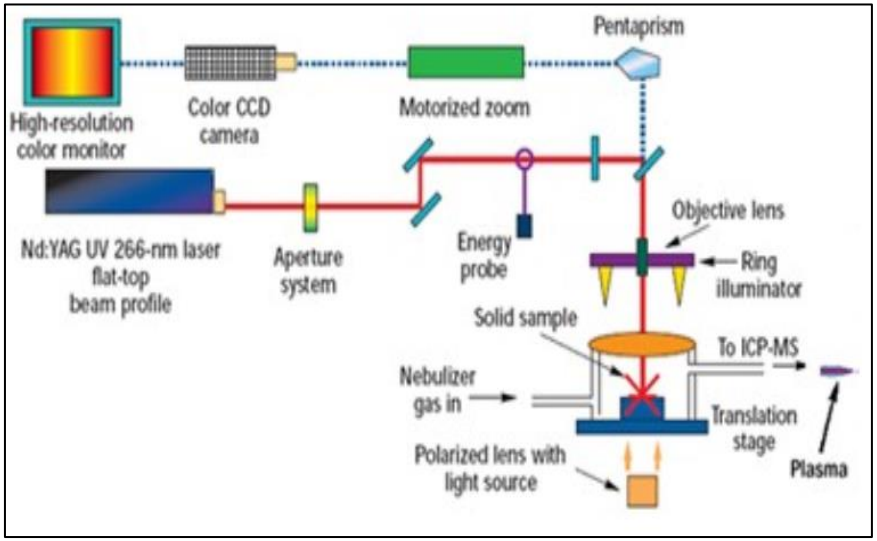


Figure 2. LA-ICP-MS diagram LA-ICP-MS Process - Laser ablation creates fine particles from the sample, which are then transported for rapid elemental and isotopic analysis.

Elemental analysis of summer truffles (Tuber aestivum)

TABLE 1: Percent composition of main metals (more than 0.01 %) found in the summer truffle.

	C	P	S	Ca	N	Si	Mg	K	Al	Na	Cl	Fe	Zn	Cu	Ti	V	Mn	Rb
%	43.20	20.91	9.91	5.43	4.79	4.48	3.07	2.90	1.82	1.18	0.60	0.50	0.39	0.15	0.10	0.07	0.05	0.01
SD	1.60	4.25	2.95	1.75	0.78	3.92	0.94	0.45	1.32	0.76	0.20	0.44	0.10	0.04	0.06	0.05	0.03	0.01

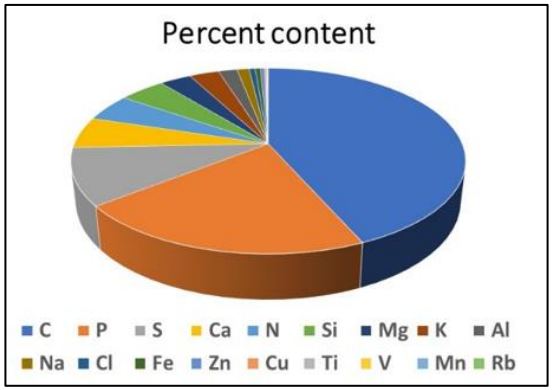


FIGURE 1: Percent composition of main metals (more than 0.01 %) found in the summer truffle.

TABLE 2: Comparison of the elemental analyses of Al, Fe, Zn and Cd (mg/kg [dry weight]) made by different methods in six samples of the summer truffle.

Analytical techniques	Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Percent
ICP-MS	Al	1575±627	811±269	640±468	847±914	393±89	490±237	1.82±1.32
PGAA		2175±189	2225±479	1725±263	1450±129	1600±115	1250±308	
ICP-MS	Fe	398±206	177±92.8	127±74	78.1±57.2	115±28.8	261±209	0.50±0.44
INAA		574±187	232±125	159±79.2	66.7±45.4	118±20.9	293±26.5	
PGAA		360±110	223±216	155±144	243±246	93.3±51.3	425±270	
ICP-MS	Zn	108±23.2	126±27.2	155±13.1	210±25.1	167±25.6	193±41.5	0.40±0.10
INAA		123±25.6	141±28.6	177±15.8	205±35	172±23.3	195±57.3	
ICP-MS	Cd	2.43±0.35	2.98±0.93	2.36±0.86	2.7±0.31	5.74±1.11	2.71±0.99	0.01±0.00
PGAA		2.07±0.3	2.44±0.7	2.07±0.72	2.13±0.24	4.43±0.84	2.13±0.79	

Conclusion:

Determination by more than one technique generally provides the results which agree within the experimental error of determination. The only exception is observed for Al, which showed systematically higher values in all samples by PGAA compared to ICP-MS.

Determination of heavy metals in the soil of the Polish side of the Białowieża Forest

Studies have been performed in collaboration with
Prof. Eu.G. Busko, Belarusian State University, ISEI BSU, 23/1 Dolgobrodskaja, Minsk, Republic of Belarus

Białowieża Forest is the last remaining primordial forest in Europe. It covers an area of over 1500 km^2 in the calm corner of Poland and Belarus.

On the Polish side, part of the Białowieża Forest exists as the Białowieża National Park, with an area of about 105 km^2. The forest is home to several types of unique birds, insects and thousands of mushrooms. It is also known as the last sanctuary of the European bison - the largest land mammal in Europe. There are also numerous old specimens of oak, elm, lime or several other representatives of trees.

For this reason, Białowieża Forest has been recognized by UNESCO as a Biosphere Reserve and World Heritage Site.

To perform quick analysis of heavy metal content is relatively difficult due to existing analytical methods being time-consuming.

Therefore, the aim of work performed in the Institute of Nuclear Chemistry and Technology in collaboration with the International Sakharov Environmental Institute of the Belarusian State University was to assess the concentration of several heavy metals in soils collected from different areas of the Białowieża National Park (Polish part) with possibly different types of anthropogenic impact.

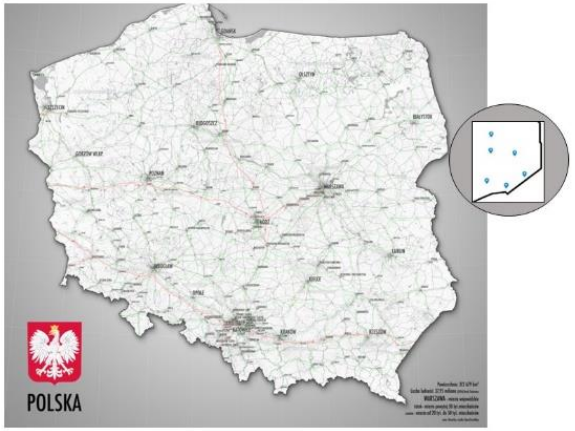


Figure 1. Sampling locations of the Bałowieża Forest soil in this study.

TABLE 1: Mean concentrations of metals in samples from different Bałowieża sites in (mg/kg) dry weight determined by the ICP-MS

Element	Mn	Zn	Pb	Ce	V	Ni	La	Nd	Cu	RE	Co	Th	U	Cd
Mean	294	24	17,53	9,79	9,55	5,82	5,18	3,94	3,09	1,41	1,33	1,32	0,59	0,11
SD	150	4	7,09	1,91	2,92	0,42	0,98	0,76	1,01		0,47	0,47	0,16	0,06

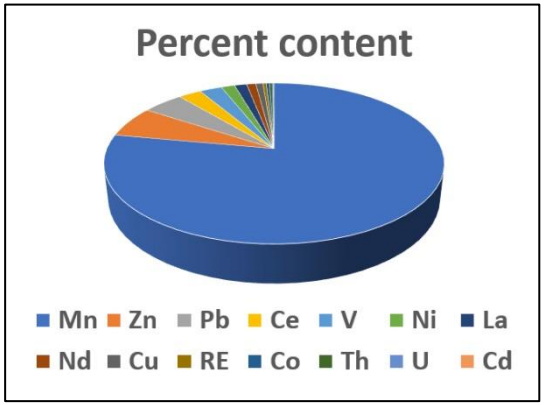


Figure 3. Percent composition of metals found in the samples collected from different sites of Bałowieża Forest determined by the ICP-MS method.

Conclusions:

Taking into account, that the determined values are low and the sample collection points are different in both studies, the ICP-MS method seems to be reliable method for analyses of the environmental samples.

Considering the fact that the determined values do not differ significantly from the results provided by Goworek et al [1], it can be assumed that the content of metals in the Białowieża Forest soil has not changed in the last twenty years.

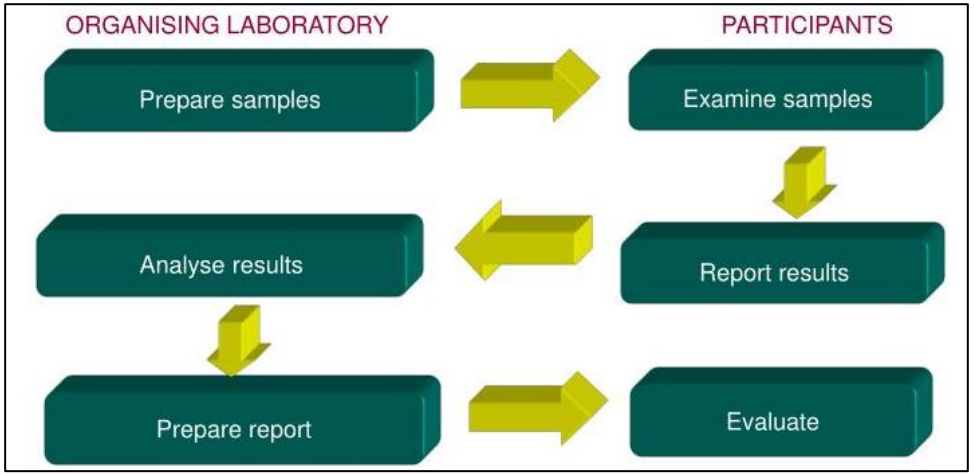
[1]. Gworek, B., Degórski, M., Brogowski, Z. (1999). *Trace metals in auto and semihydrogenic soils found in three forest site types of Bialowieza National Park*. Pol. J. Environ. Stud., 8, 305-308.

Proficiency testing on determination of radioactivity in food and environmental samples in Poland

Proficiency testing (PT), also called **interlaboratory comparison** (ILC), determines the performance of individual laboratories for specific tests or measurements and is used to monitor laboratories' continuing performance. As this term implies, proficiency testing compares the measuring results obtained by different laboratories.

In a proficiency test, one or more artifacts are sent around between a number of participating laboratories. Each laboratory measures the artifacts (e.g. an emited radiation concentration) according to a given set of instructions and reports its results to the administrator.

The results reported by each laboratory for a measurand are compared to the reference value for that measurand. The reference value can be determined in various ways. The two most common ways are to use a reference laboratory or use the average of the values reported by the participants.



Since 2004, the ICHTJ has been a provider of annual Proficiency Tests on the determination of radionuclides in food and environment organized by the NAEA. Natural matrix samples or spiked matrices have been used: ground and surface water, drinking water, milk, milk powder, flour, different dried vegetables and soil.

Up to 12 national laboratories which form radiation monitoring network in Poland participate each year in the comparison. Some laboratories from outside of the network were also invited.

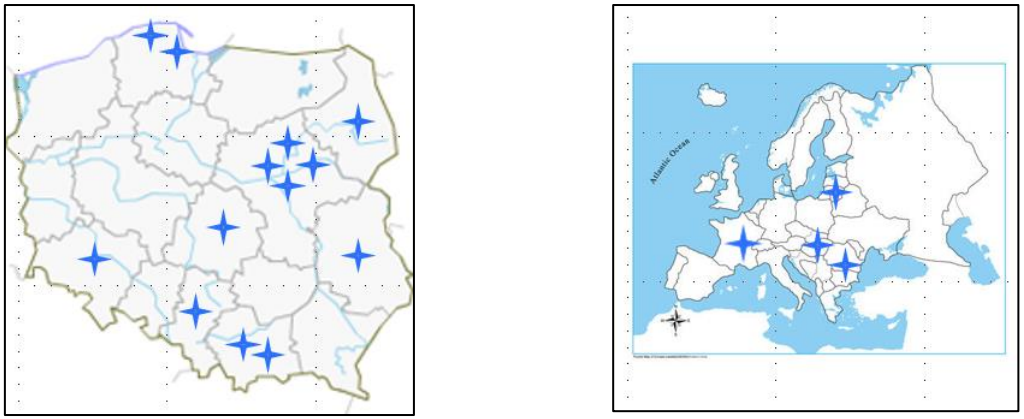


FIGURE 1: Partners from Hungary, Lithuania, Romania and France participated also in the tests.

Spiked materials	Radionuclides
water	H-3, Sr-90, Cs-137, Am-241, Ra-226 and Pu-239
milk, milk powder	Sr-90, Cs-137, Am-241 and Pu-239
flour, different dried vegetables	Sr-90, Cs-137, Am-241 and Pu-239
soil	Sr-90, Cs-137, Am-241 and Pu-239

The PTs are provided in accordance with standards: PN-EN ISO/IEC 17043:2011 and ISO 13528:2005, as well as with the IUPAC harmonized protocol published in 2006.

The PTs consist on a comparison between assigned values, determined by the ICHTJ, and the results obtained by the participating laboratories. The results provided by the participants were statistically evaluated by means of *z* and *zeta* scores as well as using the International Atomic Energy Agency (IAEA) criteria for trueness and precision.

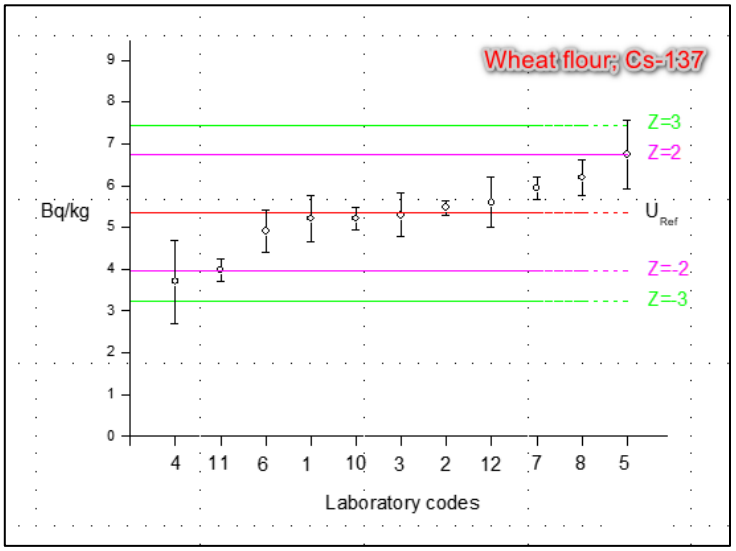


FIGURE 2: Reference value and results obtained by laboratories

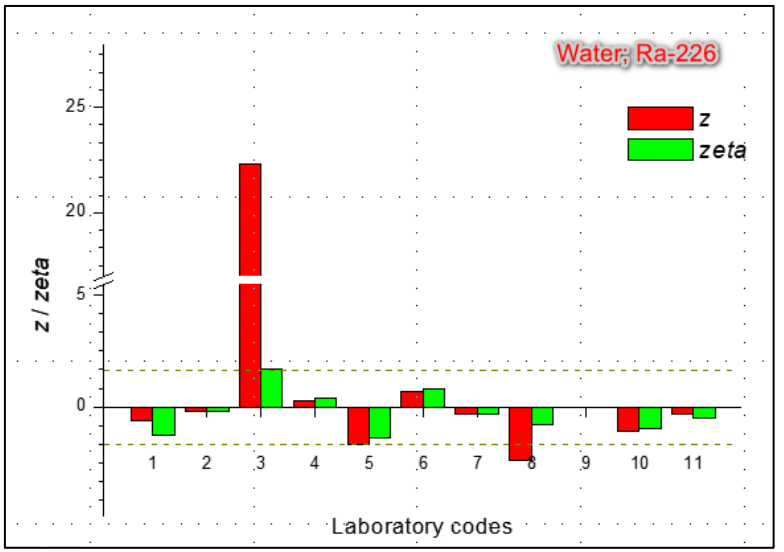


FIGURE 3: z and zeta scoring of the results

Conclusion:

All leading laboratories from the radioactivity monitoring network, participating in the PTs organized by the NAEA are high-experienced in the determination of radioactivity in environmental and food samples.

Every year, any laboratory receives a certificate of quality from the organizer.

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President
The Royal Society for the Prevention of Accidents

Institute of Nuclear Chemistry and Technology invites further laboratories to participate the PT for free of charge.

Thank you for your attention !!!