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LABORATORY ACCREDITATION AND THE CALIBRATION OF RADIOLOGICAL MEASURING TOOLS

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THE LOWER DANUBE ENVIRONMENTAL PROTECTION INSPECTORATE

Our Inspectorate is located on the left bank of the river Danube, its official area is about 6000 km^2. The lower Hungarian Danube reach is associated to it, that is 127 km long. There are 90 settlements in our district (out of which 10 are towns), the population of the district is approximately half a million.

The Inspectorate is a so-called deconcentrated state authority, a regional organisation of the Ministry of Environment Protection. There are altogether twelve Inspectorates in the country, covering all of its area, dividing it according to river catchments. The Inspectorate is authorised to comply three kind of tasks: it functions as a legal authority, as expert office and performs measuring activities. Due to this structure it meets all types of environmental problems, so as

water quality protection
air pollution prevention
soil protection
protection against harmful impacts of waste
protection against noise and vibration
protection against radioactivity

A good laboratory background is a basic necessity of specialists' decisions. This task is being met by our lab. Its activities can be divided into two main groups:

legal authority controls

and

running an environmental control (monitoring) system.

The goal of the measures performed in the lab of the Inspectorate is to get to know the polluting materials' quantitative and qualitative parameters and their dynamics occurring in the elements of the environment, and according to this, to gain the ability of evaluating the status of the environment.

Our environmental measurements are performed on soil, air, water and waste samples, and in case of radioactivity measurements of the river Danube we also examine the sediment, the fauna and flora (fish and algae).

In case of authority controls we have to determine the rate of pollutant emission of plants and factories.

On the above-mentioned environmental elements we perform:

- physical, chemical
- hydrobiological, toxicological, microbiological and
- radioactivity analysis.

A task of the laboratory is of national importance, that is the official and environmental control of the Nuclear Power Plant in Paks. The reason for this is that we are located downstream from Paks on the river Danube. The power plant - even if it's functioning properly - is emitting liquid and gas phase radioactive contaminants into the environment, that are under regulation of well-controlled emission routes and strict emission limits. That is only under these conditions that any kind of radioactive emission can be authorised, on a specified yearly level.

Our lab has got special tasks regarding this field, too, that is to execute the co-operatively organised surveys within the frame of the international agreement with the downstream states of Croatia and Yugoslavia.

The radiological measurements consist of the analysis of

- *environmental* water samples, in case of the Danube sediment, vegetation and fish samples as well, and
- water, aerosol and air samples *from the NPP*.

We perform total-beta determination, gamma-spectrometry measurements, tritium-analysis and within the radiochemical analysis Sr-90 determination on these samples.

I would like to introduce the increasing of the proficiency of the activities of our lab through the radioactivity measurements, especially through the gamma-spectrometry measurements, because these have led to accreditation and a proficiency level suitable to international standards.

ACCREDITATION AND CALIBRATION

The countries joined to the European co-operation for Accreditation of Laboratories (EAL) - so as Hungary - have undertook to establish the network of "controlled" labs, accepting common conditions and standards. The preliminary to the European accreditation system was the American GLP (Good Laboratory Practice) system, that has more decades of tradition.

Till this time in Hungary the National Accreditation System is valid, that is approaching the European system by accepting its standards one by one - so we're only joint members of this latter at present.

The European prescriptions are written down in the harmonised European standard no. EN 45001. This standard had been localised in Hungary under no. MSZ-EN 45001/1990, by the title "General conditions on the operation of analytical laboratories".

The no. ISO/IEC Guide 25 suggestion describes similar expectations. This is to appear in the near future as an international standard under no. ISO 17025, by the title "General requirements for the competence of calibration and testing laboratories ". By the time it becomes valid, in Hungary it will replace EN 45001.

I would like to point out only some of the expectations of the mentioned standard.

It, e.g. states, that the lab has to run a kind of quality-control system, that has to be appropriately designed to suit the type, the size and the quantity of the performed operations. One of the most important issues within this circumstance is "The ability of binding the measurement and calibration tools to national or international ethalons".

This expectation has extraordinary importance, because it is a basic issue in Hungary as well to use only calibrated tools in any of the legally performed measurements (so as having financial means). This is not only true to the measurement tools used in shops, for example, but to our lab measurements, too, as they can be a basis for serious environmental protection punishments. Not speaking about the fact that the customer can attack our decisions at the public court, and in such cases only the measurements performed on calibrated samples by calibrated tools are appearing as reliable.

The calibration of the measurement tools in Hungary is performed by the OMH (National Office of Measurement Affairs).

The need for the comparability and appropriate calibration was present regarding the radiological measurements from the beginning of course, since without these things some of the analysis doesn't make sense at all. Accreditation means that the already familiar and traditionally used experiences and methods are transferred to all kind of measurements.

I do not mean by this that we don't have to improve anything in the field of radiology. The accreditation indeed means a kind of methodological system, a kind of viewpoint, a laboratory philosophy, where everything is well-documented and every measured value is obviously traceable to exact standards.

Calibration itself and also standards have a certain hierarchy, so as we can speak about

international level	international standards
national level	national standards
accredited calibration labs	standards to be referred to
in-house calibration	working standards.

There's one more very important thing to speak about, that's the "certified reference material" (CRM), that has a certificate, and a defined range of uncertainty belongs to each of the certified values. The preparation procedure of the locally prepared working standards has also got to be documented.

The methodology and procedures of analysis have to be written down in an appropriate handbook or standard, or, if the lab uses a unique method, then an exact and authorised description about it is a requirement.

The regularly performed maintenance and service of the testing equipment has got to be documented as well.

In the protocol issued by the lab the estimated uncertainty of the results must be given (usually at 95 % significancy level).

The lab has to participate regularly in profile-oriented proficiency testing and comparative (inter-labs) studies, in order to ensure the quality of the measurements. For this reason, VITUKI (Bp.) coordinates the total-beta- , as well the Ministry for Agriculture coordinates the gamma-spectrometry intercalibration.

We also organise intercalibration measurements. Till now we have coordinated a tritium measurement and a Sr-90 intercalibration measurement. In such cases we also happily invite the surrounding foreign partners, *e.g.* the Ruđer Bošković Institute also has participated.

Last but not least the lab has got to have an own quality manager, who is qualified not only in the practical specialist fields but in the quality affairs, too. Her/his task is the inner audition, and management of the whole quality-insurance system. Her/his job is independent from the lab management (in our case it's directly dependent of the technical deputy manager).

The accreditation system of the lab has to be renewed on a three-years cycle, in the frame of an entirely new procedure. In the meantime the appropriate department of the NAT (National Accrediting Committee) performs audits every year.

The radiological measurements

Our lab in Baja performs radiological testing since the second half of the sixties.

In the few years' period before starting of the first block of the Nuclear Power Plant in Paks (December 1982) we performed preliminary basic radioactivity level measurements. At this time we had only some equipment. After starting the NPP Paks the increasing of the range of our tasks and the development of our equipment slowly proceeded.

We also have to speak a bit about the preparation of the samples before the execution of the analysis. In case of radiological samples this operation is often a longish one. The scheme of these preparing procedures in our lab are shown below:

Type of sample	Method of concentration	Measuring geometry
water	evaporation to dry	Al plate
sediment	drying - milling	plastic cylinder
fish	cooking - drying	plastic cylinder
algae	drying-milling-compressing	pastille
air	comprimation to 150 bar	gas cylinder, 5 L

The geometric efficiency of the radiological detectors

There are some factors that make the calibration of radiological testing equipment difficult. One of these is the determination of the geometric efficiency of the detector. As the kind of equipment vary, so do the methodology and the necessary materials.

A relatively simple task is to determine the geometric efficiency of the beta-radiation detectors.

- In case of total-beta determination we obtained the geometric efficiency by counting a non-hygroscopic potassium-compound (*e.g.* KCl or K_2SO_4).
- In case of the determination of the geometric efficiency of Sr-90 measurements, we used the method of measuring the counting speed of a known activity-concentration

Y-oxalate or $SrCO_3$ caught on filter paper, depending on the fact if the measurement had been performed in Y-90 or Sr-90 (+Y-90) equilibrium.

To determine the tritium content of water samples with application the cocktail used by us, there are certified standards provided by the OMH, or we can also use some known activity concentration tritiated water as a locally prepared source.

The accreditation of the lab in case of Sr-90 (+Y-90) had the only effect that the OMH provided us with a sealed, certified working standard measured by themselves. In case of tritium OMH regarded their own measured combination of tritiated water with cocktail as a certified one, as well.

The whole thing is more complicated in case of gamma-spectrometry:

In the beginning the working standard necessary for determination of the activity- concentration of *water samples* was prepared by ourselves, out of more liquids containing known activity-concentrations of radionuclides, in a geometry used for the measurement of the samples. Since the accreditation took place, OMH provides us with sealed, certified working standards that have the necessary geometry and surface density.

The activity concentration of *sediment samples* firstly was measured in Marinelli-dish, later in a round-shaped geometry. We performed the calibration with the help of quartz sand that was traced with a mixture of radionuclides.

In order to increase the accuracy of the measurements, a need to obtain more exact calibration sources that approach closer to the matrices of the sediment samples and their density have arisen.

Now we use traced bentonite, bentonite-Al_2O_3 and Al_2O_3, as calibration sources used for measuring the geometric efficiency of sediment samples.

Since the accreditation we have to use sealed, "not-to-open", certified quantities of the above-mentioned calibration sources, that are kept in dishes similar to the measuring geometry.

In case of *fish samples*, although their density sometimes can much differ from that of the calibration source, the traced bentonite is applied as working standard.

The gamma-spectrum of *samples of algae* was originally taken in an appropriate size laboratory cooking-glass. The density of such a sample was much varying, and the sample itself wasn't homogenous. Than we invented the use of grained, pastillised algae samples. Depending on the sample in question we prepare pastilles of the following sizes: 30x8 mm or 50xl0 mm. Good measuring geometry is a great advantage of this method, also that we can avoid pulverisation, and furthermore, the density of the pastilles is pretty close to that of the bentonite working standard.

In the beginning we prepared the calibration sources for these measurements ourselves, but since the lab is accredited, the OMH provides these "in sealed" plastic cases.

Since *air samples* are low activity-concentration and big volume, the methodology of their measuring and geometric efficiency determination would require a whole independent presentation. Shortly I would like to only mention, that when we started, we modeled the 200 bar pressure sample gas with a kind of dry vegetal (plant) sample traced by a known activity-concentration liquid. Later, together with the accreditation procedure, OMH performed a secondary calibration.

SUMMARY

I think it can be seen from the previously presented facts, that accreditation in our days is a strict requirement for a lab for its results could be accepted on international level. Accreditation itself brings to new requirements, among them some are related to the calibration of the radiological measuring equipment, that I was speaking about.

REFERENCES

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